



Università degli Studi di Cagliari

**DOTTORATO DI RICERCA**

Ingegneria Strutturale

Ciclo XXVI

**THE USE OF SARDINIAN GRANITE BY-PRODUCTS  
FROM THE SARRABUS-GERREI REGION TO BE USED  
IN ROAD PAVEMENT LAYERS**

Settore scientifico disciplinare di afferenza

ICAR /04





*James Rombi gratefully acknowledges Sardinia Regional Government for the financial support of his PhD scholarship (P.O.R. Sardegna F.S.E. Operational Programme of the Autonomous Region of Sardinia, European Social Fund 2007-2013 Axis IV Human Resources, Objective 1.3, Line of Activity 1.3.1.)*

***To my dear family for the love and support.....***





## **ACKNOWLEDGEMENTS**

First and foremost I wish to acknowledge the supervision provided by Prof. Mauro Coni and Dr. Silvia Portas, they have always been generous with time and have constantly been on hand to provide guidance and inspiration when needed. I also wish to thank them together with Prof. Cristina Trois for arranging my stay at the University of KwaZulu-Natal (Durban).

Secondly I am very grateful to Dr. Stefano Naitza, that without the enthusiastic technical input provided, I could not have completed the preliminary petrographic testing analysis.

Many thanks to the technicians of the Geotechnical Laboratory of the province of Cagliari, Mr. Gianni Vacca, Mr. Marco Diana and Dr. Carlo Piroddi for their precious technical input provided during all the test campaign. Also I would like to express my deepest gratitude to Dr. Salvatore Pistis, director of the Geotechnical Laboratory of the Province of Cagliari, for his advices and numerous discussions during the work.

I am particularly grateful to my colleagues Andreina Etzi, Alessio Contu and Franco Pigozzi, with whom I have shared many enjoyable times.

To Marta for her love, understanding and constant encouragement.

To my parents for their patient support during these years, I am grateful for the support that you have given me.

March 2014

James Rombi



## TABLE OF CONTENT

TABLE OF FIGURES .....	5
LIST OF TABLES .....	11
ABSTRACT .....	13
1. INTRODUCTION .....	15
1.1 Aims & Objectives .....	18
1.2 Thesis Layout .....	19
2. LITERATURE REVIEW .....	21
2.1 Possible Use of By-Products Deriving from Excavation Processes ..	21
2.2 European and Italian Waste Regulations.....	23
2.3 Sardinian Regional Government: Guidelines on the Possible Use of Scraps Deriving from Marble and Granite Quarries.....	27
2.4 Unbound Layers .....	29
2.5 Hydraulic Bound Layers.....	33
2.6 Bitumen Bound Layers.....	34
2.6.1 Theories and Mechanisms of Adhesion .....	34
2.7 Digital Image Processing.....	39
3. METHODOLOGY AND PROCEDURES .....	41
3.1 Introduction .....	41
3.2 Selection of the Case Study .....	41
3.3 Preliminary Characterization.....	42
3.4 Characterization of Granite By-Products for the Correct Use in Road Pavement Layers .....	43

3.5	The Use of Image Analysis .....	43
4.	SARDINIAN GRANITOIDS ROCKS AND THEIR EXPLOTATION ..	45
4.1	Introduction to the Geology of Sardinia.....	45
4.2	Sardinian Granitoids Rocks .....	47
4.3	Sarrabus-Gerrei Geological Region .....	50
4.4	Exploitation of Sardinian Granitoids.....	52
5.	CASE STUDY GRANITE BY-PRODUCTS FROM TUNNELING WORKS .....	58
6.	LABORATORY TESTS ON GRANITE BY-PRODUCTS TO BE USED IN ROAD PAVEMENT LAYERS.....	64
6.1	Material Used in Test Campaign.....	64
6.2	Environmental Compatibility .....	71
6.2.1	Leaching tests .....	71
6.2.2	Leachate analysis.....	74
6.3	Test on granite by-products to be used in unbound granular layers ..	78
6.4	Test on granite by-products to be used in hydraulic bound layers.....	97
6.5	Tests and granite by-products to be used in bitumen bond layers ...	104
6.6	Affinity between granite by-products and bitumen.....	112
6.7	The use of adhesion promoters to increase bonding between bitumen and granite aggregates.....	118
7.	DIGITAL IMAGE PROCESSING.....	121
7.1	Introduction to image processing .....	121
7.1.1	Image acquisition .....	123
7.2	Possible use of image processing to evaluate stripping test values .	124

7.2.1	Software image processing features.....	126
7.2.2	Analysis on images obtained using CAD software.....	131
7.2.3	Image analysis on real objects.....	137
7.3	Digital image processing to evaluate stripping test values .....	141
7.4	Possible use of image processing to understand parameters that influence adhesion phenomena .....	143
7.4.1	Methodology of thin section preparation .....	144
7.4.2	Image acquisition .....	147
7.4.3	Image processing and measurements .....	149
7.4.4	Data processing and results .....	152
8.	CRITICAL DISCUSSION AND CONCLUSIONS.....	161
8.1	Environmental compatibility .....	161
8.2	Unbound layers.....	162
8.3	Hydraulic bound layers.....	163
8.4	Affinity between bitumen and granite by-products.....	163
8.5	The use of digital image analysis .....	164
9.	RECOMMENDATIONS FOR FURTHER RESEARCH.....	165
	REFERENCES.....	166



## TABLE OF FIGURES

Figure 1. Flowchart for the commercialization of granite and marble .....	28
Figure 2. Aggregate shape properties .....	30
Figure 3. Contact angle between liquid and solid.....	36
Figure 4. Geodynamic context of the study area in the frame of Corsica-Sardinia anticlockwise block rotation and Liguro-Provençal basin opening .	45
Figure 5. Geological sketch map of Sardinia.....	46
Figure 6. Different regions of Sardinia .....	48
Figure 7. Simplified geological sketch map of late-hercynian granitoids of Sardinia (source Secchi and Lorrai, 2001).....	49
Figure 8. Geological map of late-Hercynian granitoids from Sarrabus area..	50
Figure 9. Abounded Granite quarry based in the locality of Monte Cilata district of Luogosanto .....	53
Figure 10. Abounded Granite quarry based in the locality of Monte Muddetru district of Calangianus.....	53
Figure 11. Abounded Granite quarry based in the locality of Stazzo Carrulu district of Calangianus.....	54
Figure 12. Commercial granite types.....	55
Figure 13. Annual granite production (source Department of Industry of the autonomous region of Sardinia).....	56
Figure 14. In red the position of the area involved by the construction site...	59
Figure 15. Satellite views of the south of Sardinia and of the area involved by the construction of the tunnels highlighting in red the length .....	60
Figure 16. Entrance of one of the main galleries and service tunnels .....	61
Figure 17. TBM without the cutting head.....	61
Figure 18. Jumbo drill in operation, source ( <a href="http://www.todini.it">http://www.todini.it</a> ).....	62
Figure 19. Mobile crusher in operation.....	62



Figure 20. Stockpile of granite deriving from TBM excavation .....	63
Figure 21. Stockpile of granite deriving from D&B excavation .....	63
Figure 22. View of the sampling points in one of the stock piles.....	64
Figure 23. Granite deriving from TBM excavation method .....	65
Figure 24. ICP-OES Perkin Elmer Optima 7000 DV (DICAAR).....	66
Figure 25. Modal compositions of the AG-1 plotted in QAP classification diagram ( <i>source Andrew Alden, geology.about.com, reproduced under educational fair use</i> ) .....	67
Figure 26. Modal compositions of the AG-2 plotted in QAP classification diagram.....	68
Figure 27. Diffractogram obtained on sample deriving from granite AG-1...	69
Figure 28. Diffractogram obtained on sample deriving from granite AG-2...	70
Figure 29. Specific gravity weighing apparatus .....	70
Figure 30. Vacuum Pycnometer.....	71
Figure 31. End to End Tumbler.....	74
Figure 32. Vacuum filtration apparatus .....	74
Figure 33. pH meter ready to perform measurements of pH and conductivity .....	75
Figure 34. Comparison of pH data values obtained from tests performed in the two campaigns.....	76
Figure 35. Comparison of conductivity data values from tests performed in the two campaigns.....	76
Figure 36. Average Particle size distribution on tests conducted on granite AG-1 .....	79
Figure 37. Average Particle size distribution on test conducted on granite AG- 2.....	80
Figure 38. Average Particle size distribution on test conducted on granite AG- 3.....	81

Figure 39. Comparison between the three granite by-products .....	82
Figure 40. Granite by-products Vs grading n°1 .....	83
Figure 41. Granite by-products Vs grading n°2 .....	84
Figure 42. Determination of the plastic limit .....	85
Figure 43. Moisture - Density relationship for the three tested granite by-products .....	86
Figure 44. Material after Los Angeles abrasion test sample AG-1 .....	87
Figure 45. Los Angeles abrasion test values on the three types of granite by-products .....	88
Figure 46. Mould, extension collar and spacer disc .....	90
Figure 47. Testing machine to perform CBR tests .....	90
Figure 48. Californian bearing ratio testing apparatus .....	91
Figure 49. Force/penetration curve case of the correction of the origin .....	92
Figure 50. Moisture-CBR relation for tests conducted on granite by-products AG-1 .....	93
Figure 51. Moisture-CBR relation for tests conducted on granite by-products AG-2 .....	93
Figure 52. Moisture-CBR relation for tests conducted on granite by-products AG-3 .....	94
Figure 53. Shaking Machine .....	95
Figure 54. Sand Equivalent test settling period .....	96
Figure 55. Soil-Cement mixture grading for Major Highways .....	98
Figure 56. Soil-Cement mixture grading for Minor Highways .....	98
Figure 57. AG-1 and AG-2 blend in order to be inside the grading for Minor Highways .....	99
Figure 58. Mixing the aggregates with water and Portland Cement .....	99
Figure 59. Samples after compaction .....	100
Figure 60. Samples removed from the moulds .....	100

Figure 61. Samples maturing for 7 days under wet sand.....	101
Figure 62. Specimens ready to perform compressive strength test .....	101
Figure 63. Sample after test .....	102
Figure 64. Compressive strength mean values .....	103
Figure 65. Comparison between HMA base layer grading and AG-2 average grading.....	105
Figure 66. Preparation of each aggregate fraction for the grading of HMA base layers .....	105
Figure 67. Comparison between HMA base layer grading, AG-2 average grading and AG-2 blend.....	106
Figure 68. Principle on which the gyratory compaction method is based....	107
Figure 69. Gyratory compactor.....	108
Figure 70. Extraction of the specimen from the mould .....	108
Figure 71. HMA open grade layer and the AG-2 blend obtained.....	110
Figure 72. Open-graded HMA specimen.....	111
Figure 73. Silica content for some common aggregates .....	112
Figure 74. Un-compacted coated aggregates .....	114
Figure 75. Specimens at constant temperature of 40°C.....	114
Figure 76. Basalt aggregate - Figure 77. Aggregate from crusher .....	115
Figure 78. Granite from TBM - Figure 79. Granite from TBM .....	115
Figure 80. Basalt Aggregates - Figure 81. Granite from crusher .....	116
Figure 82. Granite from TBM - Figure 83. Granite from D&B .....	116
Figure 84. Adhesion promoters function between bitumen and acidic aggregates.....	118
Figure 85. Granite by-products with no adhesion promoter.....	119
Figure 86. Granite by-product with adhesion promoter (Iterlene IN 400-S) .....	119
Figure 87. Granite by-product with adhesion promoter (Iterlene PE 31-F ) .....	120
Figure 88. Digitalization of a continuous image.....	122

Figure 89. Image analysis major steps of processing .....	124
Figure 90. Image Pro Plus brightness, contrast and gamma dialogue box. ..	126
Figure 91. Calibration dialogue box .....	127
Figure 92. Count and measure dialogue box .....	128
Figure 93. View of Pixcavator commands.....	129
Figure 94. Main features .....	130
Figure 95. Images designed using cad software .....	132
Figure 96. Multiple circles.....	134
Figure 97. Image different diameters.....	135
Figure 98. Image analysis using Image-Pro Plus with images at different resolutions .....	136
Figure 99. Sample holder .....	137
Figure 100. Sample ready for image analysis.....	138
Figure 101. Software image calibration.....	139
Figure 102. Object search .....	139
Figure 103. Before and after performing stripping test .....	141
Figure 104. Image analysis to search black colour.....	142
Figure 105. Area covered by bitumen for each aggregate eliminating the rest .....	142
Figure 106. Silicon sample holders.....	144
Figure 107. Epoxy resin and hardener .....	145
Figure 108. Specimen under vacuum.....	145
Figure 109. Thin sections.....	146
Figure 110. Light optical microscope .....	147
Figure 111. Overview of axioplan microscope.....	147
Figure 112. Digital microscope camera .....	148
Figure 113. Thin section image enlarged x25.....	148
Figure 114. Thin section in crossed (A) and plain-polarized light (B).....	149

Figure 115. Bitumen film thickness measurement .....	150
Figure 116. Progressive measurement of aggregate morphology.....	151
Figure 117. Mineral composition of the aggregate .....	151
Figure 118. Thin section with aggregates deriving from D&B .....	152
Figure 119. Granite by-product deriving from D&B subjected to stripping test at 40°C .....	153
Figure 120. Relation between the aggregate roughness and bitumen film thickness .....	154
Figure 121. Granite by-product deriving from D&B subjected to stripping test at 40°C .....	155
Figure 122. Relation between the aggregate roughness and bitumen film thickness section n°1 .....	156
Figure 123. Relation between the aggregate roughness and bitumen film thickness section n°2 .....	157
Figure 124. Granite by-product deriving from D&B subjected to stripping test at 40°C .....	158
Figure 125. Relation between the aggregate roughness and bitumen film thickness aggregate labelled n°3 .....	159
Figure 126. Basic rock deriving from D&B subjected to stripping test at 40°C .....	159
Figure 127. Relation between the aggregate roughness and bitumen film thickness aggregate labelled n°4 .....	160

## LIST OF TABLES

Table 1. Chemical composition of granite by-products deriving from TBM (AG-1) and D&B (AG-2).....	66
Table 2. Modal composition of representative AG-1 and AG-2 granites.....	67
Table 3. Maximum permissible concentration of elements.....	72
Table 4. List of Elements according to D.M. n. 161/2012.....	73
Table 5. pH and conductivity mean values deriving from tests of each campaign .....	75
Table 6. First campaign set of test results on elements imposed by the LG.D. n. 152/2006.....	77
Table 7. Second set of tests on elements imposed by D.M. n. 161/2012 .....	78
Table 8. Standard Deviation of the three types of granites analysed.....	81
Table 9. Upper and lower limit for the two types of gradings.....	82
Table 10. Optimum water content and dry density values for the three types of granite by-products.....	86
Table 11. Los Angeles abrasion test values .....	88
Table 12. Sand Equivalent Test Values .....	96
Table 13. Gradings imposed by C.N.R. 29/72 .....	97
Table 14. Mean Compressive strength values related to cement and water content .....	103
Table 15. Upper and lower limits for HMA base layer .....	104
Table 16. Upper and lower limits for Open-graded HMA layers.....	109
Table 17. Values obtained by visual observation .....	116
Table 18. Stripping values for the three tested samples.....	120
Table 19. Referable parameters.....	122
Table 20. Errors blacks circles and white background .....	132
Table 21. Errors white circles and black background.....	133

Table 22. Coloured circles white background .....	133
Table 23. Coloured circles black background.....	133
Table 24. Mean values obtained.....	135
Table 25. Mean values obtained.....	136
Table 26. Error made during the image analysis .....	140
Table 27. Results from image analysis .....	143
Table 28. Measurements conducted on the aggregate n°1 .....	153
Table 29. Measurements aggregate n°2 section n°1 .....	155
Table 30. Measurements aggregate n°2 section n°2 .....	156
Table 31. Measurements of aggregate labelled n°3 .....	158
Table 32. Measurements of aggregate labelled n°4 .....	160

## **ABSTRACT**

In Sardinia abandoned granite by-products stockpiles deriving from different excavation and processing methods are becoming an environmental and economic concern. Currently, these materials derive mainly from the ornamental quarry industry, very active in the region since late 1900's, but also from civil engineering works consisting of tunnels, dams and other excavation projects. The storage of granite by-products in such great amount and for long periods produces damage to the environment, in terms of alterations of the landscape that can be temporary but in several cases permanent when waste deposits become part of the landscape surrounding, subtracting large extensions of land to more important purposes such as agriculture. Also there is the visual and air pollution problem due to the suspension of fine particles in the air that these materials can produce when exposed for years to environmental climate conditions.

The possibility of using of such materials that are already been extracted and that for them energy and therefore CO<sub>2</sub> emissions has already been spent, would decrease the use of other natural aggregates that have yet to be excavated instead. Reducing the exploitation of non-renewable natural resources and limiting consumption of energy and CO<sub>2</sub> emissions. In order to limit and in some cases solve some of these problems this thesis takes in to account the possibility of using granite by-products for road construction purposes. The possibility of using these materials, that in many cases are considered a waste, as a valid alternative to other aggregates that normally are used in pavement layers could decrease road construction costs.

In this research granite by-products from the Sarrabus-Gerrei region were studied to evaluate the physical and mechanical properties to assess the possibility of using them to their fullest extent in both unbound and bound pavement layers. Three types of granite by-products deriving from the same mother rock but subjected to different processes were studied. Two resulting from: Drill and Blast (D&B) and Tunnel Boring machine (TBM) excavation methods. The third material was obtained from the crushing of the larger blocks obtained during D&B excavation. The work reported herein describes test conducted in order to determine the best suitable application according to the type of layer in which use such material, evaluating the possible variations in terms of physical and mechanical characteristics that granite by-product performed when subjected to different processes.



A preliminary characterization was conducted evaluating the environmental compatibility of such materials, based on Italian regulations on the use of excavated earth and rocks. The results of such tests were important to assess and determine the possible application in road pavement layers. Firstly, unbound layers were studied, analyzing aggregate properties and performing tests on different mixes. Particular attention was given on the bound layers where both hydraulic and bitumen binders were studied. Soil-cement mixes were prepared using different percentages of Portland cement performing different. Then Hot Mix Asphalt (HMA) samples were analysed in order to evaluate the affinity between granite and bitumen. Difficulties were emerged using this type of aggregates in HMA layers due to adhesion problems between granite and bitumen. Stripping tests were performed and also attention was focused on the adhesion phenomena. In order to better understand such phenomena and evaluate and measure potential parameters, digital image processing was conducted. Good values for the unbound layer, in which granite by-products were tested, were measured. Furthermore, the use of such materials for cemented treated layers was evaluated obtaining as well good results.

Keywords: granite by-products, road industry, pavement layers, adhesion.

## **1. INTRODUCTION**

Granite like marble is used all over the world mainly for ornamental and architectural purposes; these types of natural stones are considered the most precious ones.

In Sardinia the quarrying of granite for ornamental purposes started as an organized trade in 1870 and has grown ever since becoming an important economical resource for the region. There are 11 types of commercialized granite in Sardinia that differ not only in colour but in some cases for physical and mechanical properties (Natural stones from Sardinia, 2002). Considering the ornamental quarry industry that is mainly concentrated on the extraction of granite and marble there are over n°730 quarries dislocated in Sardinia of which n°277 are still operating according to the last survey dated 2007 with an estimated annual production of 152.000 m<sup>3</sup> (Quarry office, 2007).

Commercial granite is subjected to many processes like excavation, cutting, polishing and grinding. The yield wastage ratio during processing varies between 65% and 50% depending on the type of granite. It is very important to understand and evaluate what must be considered a waste or what can be considered a by-product evaluating their possible application. Assuming that in the next 10 years Sardinian production remains at the actual average, there will be 1,5 million m<sup>3</sup> of suitable granite wastes plus other 40 million m<sup>3</sup> of large and medium size shapeless granite by-products already stockpiled belonging to the past granite query industry. All this stockpiled material is subtracting and will continue to subtract land if a solution isn't found.

In recent year's studies on the possibility of using of what remains from the extraction and processing phase of this type of rocks is becoming a challenging task. The possibility of giving new life to such materials can become an environmental benefit but also be a valid alternative to other natural aggregates that normally are used in the construction industry. Countries like America, Brazil, China, India and Turkey in which large quantities of granite are extracted each year are focusing the attention on this problem. The need to characterize and evaluate physical and mechanical performances of such materials is important to establish the potential applications highlighting limitations and advantages.

Granite has many positive characteristics it is solid, resistant, surface with large texture depth and is one of the hardest rocks with high resistance to wear. Granite also has many physical properties such as resistance to polishing that are superior to those of basalt and

limestone which are presently widely used in road pavements (Li, 2005) and (Zhou, 2003). Although all these characteristics are in favour for its use in the road industry, it is important to evaluate if the processes from which granite by-products derive do not modify some of these positive features that will unable or limit its use.

Recent studies conducted in Brazil, India and Turkey focused the attention on using granite fines deriving from cutting and polishing, to be used as fillers in Asphalt Concrete. In Brazil the possibility of using granite filler was studied conducting tests on evaluating the absorption of cement asphaltic on granite fines and also mechanical resistance tests were performed on asphalt mixtures. It was verified a great potential to use granite fines instead of basalt rock in asphalt production (Ribeiro et al., 2008). Also in India studies have been conducted on both granite and marble dust to be used as filler in Asphalt Concrete. The adding of marble and granite dusts as filler to Asphalt Concrete can produce properties comparable to the conventional asphalt concrete mixes (Chandra and Choudhary, 2012). In Turkey several researches take in to account the possibility of using granite and marble by-products considering the use of fines materials mainly deriving from sawmills.

Although the potential use of granite by-products in road industry is becoming a more common field of research it is important to be able to evaluate all the physical and mechanical characteristics that this type of stone can achieve considering both the nature of the material and more important the processes to which this material is undergone. For this reason this research takes in to account the study of Sardinian granite in order to replace common aggregates normally used in the road industry, verifying the potential application in all the pavement layers starting from the unbound ones and finishing with the bound upper layers. Before conducting any laboratory tests and any type of evaluation on the results it was important to establish, considering Italian regulations governing the use of excavated earth and rocks, if the waste deriving from the processes of Sardinian granite could be considered a by-product and in which terms. For this reason in this research there is a short part dedicated to Italian regulations on the use of excavated earth and rocks.

The research focused the attention on a case study considering three types of granite by-products deriving from the excavation of three tunnels for a total length of 4.1 km. Each granite by-product derived from the same type of mother rock but was subjected to different processes. Two by-products derived from different excavation techniques: material deriving from a Tunnel Boring Machine (TBM) and from Drilling and Blasting

(D&B). While the aggregates deriving from the TBM were already in a range of size suitable for the road industry, the aggregates deriving from D&B were very heterogeneous in term of size. For this reason the larger blocks were reduced using a mobile crusher.

Each material was tested to verify the more suitable application in terms of performances. First the use of granite by-products for unbound layers were studied performing laboratory tests to evaluate the influence of the physical properties of the aggregate grains, such as size, shape, surface texture, mineralogy and resistance to fragmentation.

Also the bound layers were studied performing tests on soil-cement mixture specimens evaluating compressive strength values. Special attention was given to the bound layers where bitumen binders are used. It is known that granite has adhesion problems with bitumen, adhesion is a complex phenomenon depending on many variables, this is normally related to the chemical composition of granite but also due to other factor that can influence such phenomena for example surface texture, porosity and shape of the aggregates. In this research digital image processing was used to be able to evaluate parameters that influence adhesion analyzing planar images of thin sections of bituminous mixture, in which the aggregate is covered by thin layer of bitumen. Only recently this type of technique is being used to study asphalt structures, other attempts have been made using X-ray computer tomography but this type of technique results expensive and has potential health risks.

## **1.1 Aims & Objectives**

The aims of the research can be summarized as follows:

- Verify the potential applications of Sardinian granite by-products for road construction purposes.
- Verify if different excavation techniques modify physical and mechanical behavior of granite by-products when used in road pavement layers.
- Evaluate the performances and compare them with those of natural aggregates that normally are used in road construction processes considering the best applications for each pavement layer.

Specific objectives of the research:

- Describe what is currently known on the use of granite by-products for their use in the road construction industry.
- Evaluate environmental compatibility to verify the release of hazardous elements
- Evaluate the potential applications for all the layers of the pavement.
- Analyze what is known as a critical application of granite and bitumen, analyzing stripping problem.
- Evaluate the potential use of static image analysis to better understand, evaluate and determine parameters that influence the bonding between granite by-products and bitumen.

As will be discussed in detail in the methodology Chapter, the research was divided in three main phases. In the first phase the granite by-products chosen for the research were analyzed for a preliminary characterization and their environmental compatibility was determine in order to evaluate any release of hazardous elements. In the second phase specified tests were conducted to use such materials in unbound and bound pavement layers. And in the last phase special attention was paid on better understanding the adhesion problem between granite and bitumen verifying the possible use of digital image processing technique.

## **1.2 Thesis Layout**

This thesis is organised with the following chapters:

### **Chapter 1 - Introduction**

The current chapter introduces the topic of study introducing the general aims and the specific objectives of the research.

### **Chapter 2 - Literature Review**

Introduces what is known in terms of concepts and theories considering the gaps in knowledge.

### **Chapter 3 - Methodology and Procedures**

Expresses the type of approach and the ideas that have been conducted in order to achieve the aims proposed.

### **Chapter 4 - Sardinia Granitoids and their Exploitation**

Introduction on the geology of Sardinia considering the exploitation of ornamental stones focusing the attention on granite.

### **Chapter 5 - Case Study of Granite By-Products from Tunneling Works**

Describes the choice of considering a case study to perform this research introducing useful information's on the considered case study.

### **Chapter 6 - Laboratory Tests on Granite By-Products to be used in Road Pavement Layers**

Explains the type of laboratory tests conducted and the results obtained in both unbound and bound pavement layers.

### **Chapter 7 - Digital Image Processing**

Introduces the image processing technique method and the possible application of this technology as a valid support to better understand and evaluate the parameters that influence the adhesion phenomena between granite and bitumen.

## Chapter 8 - Critical Discussion and Conclusions

The main results from the research are summarized evaluating if all the aims of the research have been achieved discussing possible ideas for future applications to conduct further studies.

## Chapter 9 - Recommendations for Further Research

This chapter consists on providing topics for future research, following the research limitations that have been identified in the present work.

## **2. LITERATURE REVIEW**

### **2.1 Possible Use of By-Products Deriving from Excavation Processes**

Annually large quantities of natural resources deriving from mines and queries are extracted, in order to fulfill the growth in industrial production. This high volume of extraction has led to a consequent increase in waste materials which has adverse impacts on the environment. The ornamental quarry industry produces large quantities of waste materials deriving from different processes to which the stones that are being extracted are subjected. Quarrying operations for ornamental purposes involve many different phases that can be summarized as follows: pre-production operations; primary cuts; secondary cuts and finishing of blocks (Gazi et al., 2012). Also other excavation methodologies and techniques can produce waste, especially in the construction industry where large quantities of material is removed for the realization of infrastructure like tunnels and dams. For this reason many countries and international establishments have been working for new regulations on how to minimize and reuse the generated waste, it is a priority to think about a systematic reuse of the rock wastes as by-products. Almost more than 89% of the international quarrying activity is concentrated in nine countries, each of them producing more than 2 million tons of natural ornamental stones per annum China, Italy, India, Iran, Spain, Turkey, Brazil, Greece and Portugal (Founti et al., 2010). Carbonatic rocks for the production of ornamental stones are quarried in the great majority of nations in the world. Granite and marble constitute the main products amounting to almost 70% of world production. In this scenario Italy is one of the leading countries for the commercialization of granite, making Sardinian granite to account for 75% of Italy's total granite output. Each year according to the Regional Industrial Ministry, Sardinia has an annual production of 152.000 m<sup>3</sup>. The yield wastage ratio during mining processing is low for the monzogranite type 65% and higher for the other trade types 50%. All this material up to now lays abandoned without any consideration on its possible applications. Some of the major producing countries are conducting researches on the possible use of what remains from the many processes to which granite is subjected before reaching commercialization. Mainly the attention is being focused on the use of the fine particles that due to their dimensions can become threatening for human and animal health. The use of such material



is being studied mainly to be used as filler. In Brazil the possibility of using granite and marble dust deriving from the cutting of the larger blocks has been evaluated. The main objective of the research was to evaluate the incorporation of these fines as a mineral aggregate in asphalt formation and evaluate the performance and compare it with that of basalt rocks (Ribeiro et al., 2008). Tests have been conducted to evaluate the absorption between cement asphaltic and granite fines by measuring the reduction in concentration of toluene solutions of these compounds after contact with the minerals. Also tests were performed to evaluate the mechanical resistance of asphalt mixtures. From these tests a great potential use of granite fines instead of basalt rock for asphalt production emerged. Similar research was performed in India where the use of marble and granite dust as filler in asphalt concrete was evaluated. Dynamic shear rheometer and ring and ball softening point test were used to evaluate the effect of different percentages of these industrial wastes on properties of asphalt-filler matrix. Marshall stability parameters, permanent deformation from static creep test and Tensile Strength Ratio (TSR) were also evaluated (Chandra and Choudhary, 2012). Such tests showed that the adding of marble and granite fine particles to asphalt concrete can produce properties comparable to the conventional asphalt concrete mixes with stone dust as filler.

Also the possibility of using granite and marble sawing power wastes in the production of bricks was studied (Dhanapandian and Gnanavel, 2010). In Turkey studies on how to be able to recycle granite and marble wastes deriving from ornamental quarrying activities is being developed considering both coarse and fine aggregates. The durability of concrete using granite and marble wastes with a maximum nominal size of 19 mm was studied. The results of this study showed that the marble and granite waste aggregates can be used to improve the mechanical properties, workability and chemical resistance of the conventional concrete mixtures (Binici et al., 2008). All these studies demonstrate that there is a scientific interest to evaluate the possible applications of this type of aggregates in many parts of the world focusing the attention on using the finer particles as filler.

## 2.2 European and Italian Waste Regulations

The Italian regulation on the use of excavated earth and rocks is in continuous modification. In recent years Italian regulations that deal with excavated earth and rocks have changed in order to fulfill European law.

The following directive is the key role to which all European countries have to fulfil **Directive 2008/98/EC of the European Parliament and of the Council of 19 November 2008** which sets down the fundamental principles and rules when considering the management of waste. In order to better understand subject matter, scope and definitions some articles of the European Directive 2008/98/EC will be reported (Official Journal of the European Union, 2008):

Chapter 1, Article 1:

*“This Directive lays down measures to protect the environment and human health by preventing or reducing the adverse impacts of the generation and management of waste and by reducing overall impacts of resource use and improving the efficiency of such use”.*

In this Directive the following definitions are applied:

Chapter 1, Article 3:

*“Waste means any substance or object which the holder discards or intends or is required to discard”*

Chapter 1, Article 5:

*“By-product a substance or object, resulting from a production process, the primary aim of which is not the production of that item, may be regarded as not being waste but as being a by-product only if the following conditions are met”:*

- (a) further use of the substance or object is certain;*
- (b) the substance or object can be used directly without any further processing other than normal industrial practice;*
- (c) the substance or object is produced as an integral part of a production process;*
- (d) further use is lawful, i.e. the substance or object fulfils all relevant product, environmental and health protection requirements for the specific use and will not lead to overall adverse environmental or human health impacts.*

Based on the articles previously reported, the European Directive **2008/98/EC** introduces measures to determine the criteria by which specific substances or objects can be

considered a by-product and not a waste. All the European Community States have to implement such directive informing the European Commission on their progress.

Italy on the bases of the European Directive is performing regulations and legislations in order to implement totally such Directive. The different sources of law that will follow are directly addressed to implement the European Directive.

Before performing a legislative framework that will be reported in a chronological order it is important to clarify that in the Italian system there are (Perez et al., 2013):

a) *primary sources of law*: the Law (L.), the Legislative Decree (LG.D.) and the Decree Law (D.L.);

b) *secondary sources of law*: Regulations, taking the form of Decree of the President of the Republic (D.P.R.) or Ministerial Decree (D.M.).

One important Decree to consider was the so-called Ronchi Decree (**Legislative Decree 22 of 5 February 1997**), which was immediately subject to two substantial corrections and has constituted the basis for innumerable detailed provisions. Italy in various occasions, specifically referring to waste, has been subject to infringement proceedings for failing to incorporate or incorrectly incorporating the Community provisions. For example among the various penalties imposed on Italy, see Court of Justice, 18 December 2007, C-263/05, *Commission/Italy* (on the notion of waste).

**LG.D. n. 22/1997**: is the first legislation that truly considers environmental issues implementing the Directive 91/156/EEC on *waste*, Directive 91/689/EEC on *hazardous waste* and Directive 94/62/EC on *packaging and packaging waste*. Also in Annex I is stated and reproduced the *European Waste Catalogue (E.W.C.)*. Still this Decree was subjected to corrections and was replaced by LG.D. n. 152/2006.

**LG.D. n. 152/2006**: the so called “Environmental Code” it covers many environmental sectors, among which waste and also the European Parliament directive 2008/98 was incorporated into the Italian legal framework by Part IV of the Environmental Code (Legislative Decree 152 of 3 April 2006) as amended by **Legislative Decree 205 of 3 December 2010** (this covers some sixty articles, Articles 177-238), to which the regional legal requirements must also be adapted (Article 177).

It repealed the former National Waste framework Law, legislative decree 22/97 that transposed three of the main EU directives on waste: the European Waste Framework

Directive 75/442/EEC, as modified by Directive 91/156/EEC, the Directive on Hazardous Waste 91/689/EC and the Directive on Packaging and Packaging Waste 94/62/EC.

**D.M. n. 161/2012**: regulates terms and conditions at which *excavated materials* may be reused as *by-products* and thus managed as *non-waste*, the discipline introduces the regulatory definition of “normal industrial practice” and does not provide a simplified procedure for small sites.

**L. n. 98/2013**: this law derives from the so-called “il Decreto del Fare” (Decree of Doing), that doesn’t introduces substantial changes to the previous D.M. 161/2012. Main issue is that who deals with excavation materials and would like to use it as a by-product must clearly state that possession of certain "requirements": certainty of use, certainty of the destination site, not exceeding the Threshold Concentration of Contamination, no health risk, no preventive treatment).

One of the fundamental problems that Italian legislators had to face was the definition of waste; this was essential information that the inclusion of a substance or object in this category had an important consequence in terms of how it was managed. Waste as reported in the European Directive **2008/98/EC** and also reported in Article 183(1) of Legislative Decree 152/2006 has a precise meaning (De Leonardis, 2011).

To better understand the notion of **by-product**, it is important to clarify that industrial production creates unwanted secondary products that can be reused. Initially, it was thought that these secondary products should be classified as waste.

Following the *Palin Granit* decision in 2002 (Court of Justice, 18 April 2002, C-9/00), in which Supreme Administrative Court led to the conclusion that all materials produced as a by-product of a main process should not be residue products, but rather by-products which the operator must use in a financially sustainable manner, rather than disposing of them. However, this applies only to material which does not have to be altered or processed in any way before its subsequent use.

While in an initial phase the notion of by-product was limited to scenarios in which the company used the by-product in the same production of process from which such material

derived, the notion was later broadened to include the scenario whereby the same company used the by-product in a different production process.

In this led to define the current definition of by-product, which means any substance or object that is generated on a secondary basis by a production process and that can be used, without any specific processing, in the course of the same or another production process.

## **2.3 Sardinian Regional Government: Guidelines on the Possible Use of Scraps Deriving from Marble and Granite Quarries**

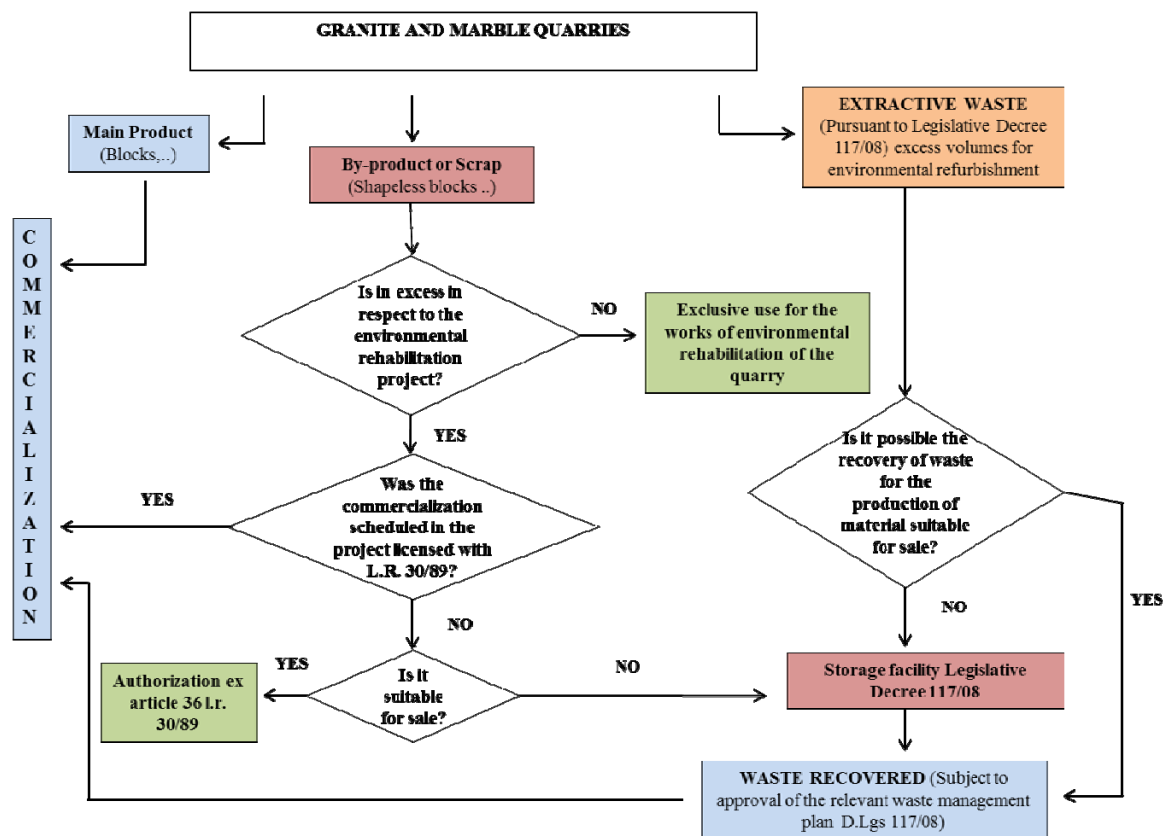
The Sardinian regional government in 2012 has introduced preliminary guidelines on the possible use of scraps deriving from marble and granite quarries to be used as aggregates in the road industry. The purpose of this document derives from the encouragement by the Public Administration of the use of goods and services that reduce the exploitation of natural resources, production of waste, the emission of pollutants, hazards and environmental risks to health and the use of renewable energy sources. The Regional Government of Sardinia is following the European Commission guidelines for the correct practice of the Green Public Procurement (GPP). To better understand what GPP is this is a short description: *a process whereby public authorities seek to procure goods, services and works with a reduced environmental impact throughout their life cycle when compared to goods, services and works with the same primary function that would otherwise be procured. GPP is a voluntary instrument, which means that Member States and public authorities can determine the extent to which they implement it* (European Commission, 2012).

The guidelines that have been introduced in Sardinia are intended to serve as a reference to engineers, construction managers and security managers allowing them to operate in terms of environmental sustainability, improving the quality of life in accordance with the following main principles:

- reducing both the effects of the exploitation of non-renewable natural resources and the environmental effect of transporting aggregates with the aim of reducing pollutant emissions
- the reduction of waste production

Aiming at designing and constructing towards a more sustainable and environmental friendly way. With this document there is a first approach on the concern that this material deriving from the processes of the marble and granite ornamental stone industry can be considered a precious resource and no longer a waste. The introduction of a methodology in which is stated the types of materials that can be used, where to use it and in which terms. This is a first step that will facilitate the introduction of scrap materials in Sardinian road pavement industry.

In Figure 1 is reported the flowchart that summarizes the methodology that must be followed to commercialize scrap and by-products deriving from granite and marble ornamental stone quarries (Regione Autonoma della Sardegna, 2012).



**Figure 1. Flowchart for the commercialization of granite and marble**

At the moment these types of materials are accepted to be used in subgrade layers and in the body of embankments. No specific laboratory tests are requested in addition to those that normally are conducted on aggregates that will be used in these pavement layers. While specific test values have to be obtained ones the material has been laid in place. That is performing static plate load tests in accordance with the CNR 146/92 specifications. For this reason specific guidelines that consider specific tests to be conducted and also values to be obtained must be drawn due to the heterogeneity of the material that we are dealing with and the alteration that can influence the aggregates depending on the type of processes to which it has undergone during excavation.

## **2.4 Unbound Layers**

Unbound aggregates have formed the basis of all engineered pavements since engineering began (Dawson, 1994). The properties of aggregates used in unbound base and sub-base layers are important in the performance of the pavement system in which they are used (Hall, 1998). Life and cost maintenance of both flexible and rigid pavements depends on the good or poor performance of the unbound granular layers.

In unbound granular layers the interaction between the single stones (mostly large) combined with the influence of the finer aggregates particles and the amount of water present in the mix guide the layer performance and behavior.

In order to obtain the more suitable aggregate interlocking combination proper aggregate selection is necessary to achieve the desired performance. Aggregate gradation is one of the more important characteristics that influence the stiffness, shear strength, and permeability combined with aggregate "geometric" characteristics of shape, angularity and surface texture. All these properties are fundamental elements that contribute to the structural adequacy and capacity of the layer.

The possibility of not achieving such performances can result in several types of road failures. In flexible pavements the failures (resulting from poor performance of granular bases) are manifested as rutting and deformation, fatigue cracking, longitudinal cracking, depressions. In rigid pavements the failure distresses are recognized as pumping, faulting, cracking, corner breaks, and, to a lesser degree, fatigue cracking (Hall, 1998).

It is possible to analyze the unbound layers by a macroscopic and microscopic point of view. Analyzing the macroscopic scale aggregate performs by being stiff, resistant to permanent deformation, while in the microscopic scale the inter-particle contact combined with the single particle roughness and micro-texture, have an important influence on the mechanical behaviour of the aggregates. Each of these characteristics will be discussed as follows.

Stiffness, or resilient modulus, of the aggregate layer is the mechanism by which the layer spreads load down from the high localized stresses imposed by vehicle tires to the level which the subgrade can tolerate without undergoing excessive deformation. Thom and Brown investigated the influence of grading and dry density on the aggregate behaviour performing repeated loading triaxial tests.



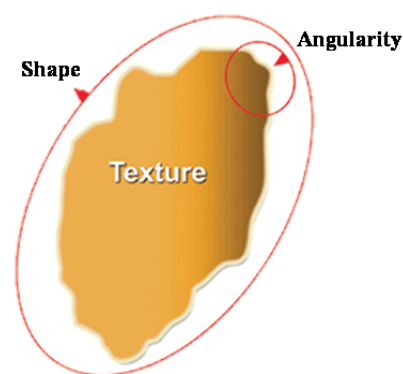
They observed that while the resilient properties seem to be ruled by the material itself permanent deformation and shear strength can be considered to be highly influenced by geometric factors such as grading, void ratio, degree of interlocking and stone surface (micro-texture) friction angle between the particles (Thom and Brown, 1988). The explanation of behavior by the micro-texture and particle roughness is likely to be complex but would include an assessment of the behavior of the points at which stones touch. Here there may be rolling, slippage and wearing of the asperities of the stones.

Packing also has a major influence. It will be controlled both by the grading and by the density. Hecht studied the geo-mechanical properties of an aggregate (density, elastic modulus.) comparing different grading's and referring to some classical packing models (Hecht, 2000, 2004).

A change in grading and density can be expected to result in a different value of the coordination number (number of contact points between a particle and its neighbours) leading to a different stress distribution over the particles surface: a coarse aggregate consisting of grains of the same size with a small fine fraction will load each particle with higher tensile stresses than if the same particles were conveniently surrounded by an appropriate number of smaller particles.

To be able to evaluate the mentioned phenomenon's certain particle properties are important. Shape properties are shown in **Errore. L'autoriferimento non è valido per un segnalibro.** and also reported together with other aggregate properties in the list that follows:

1. gradation
2. shape and particle texture
3. particle angularity
4. mechanical and chemical durability
5. specific gravity
6. toughness
7. petrographic classification



**Figure 2. Aggregate shape properties**

Despite the material's physical properties, the studies conducted by Proctor and others (Semmelink, 1991) were able to evaluate that factors such as the moisture content, the magnitude and the manner in which compaction was being produced, as well as the reactive support of the underlying material during the compaction process all had an important influence on the results that could be achieved.

Resilient behavior of unbound granular materials has been found to be very sensitive to moisture content, density and stress level to which the material is exposed. In this field of study relationships between resilient behavior and other material properties have been conducted by Thompson and Robnett (Thompson and Robnett, 1979) that studied the resilient properties of Illinois soils.

Considering this matter also Rada and Witczak presented an evaluation of the variables that influence resilient modulus response of granular materials (Rada and Witczak, 1981).

Parameters such as moisture content and density will be introduced.

Moisture content:

Water is a polar material, which means that the molecules have a definite positive and negative direction, allowing the molecules to combine with the minerals present in the aggregate surface. Water due to the phenomenon of capillarity tends to migrate into the layers pore system if the pores are small enough; this is related to the grain size distribution of the material and the amount of fines. The presence of a moderate amount of water provides benefits in terms of strength and the stress and strain behavior. Studies have been performed on the effect of water on the resilient modulus. For instance Hicks and Monismith reported an apparent reduction in resilient modulus with increasing water content (Hicks and Monismith, 1971). Also Barksdale and Itani observed a significant reduction in resilient modulus for four materials tested upon soaking (Barksdale and Itani, 1989). Also it was noted that the effect of water on the resilient properties was more significant in well graded material in which high amount of fines were present (Raad et al., 1992). In summary it is possible to reach an overall conclusion stating that the resilient modulus is reduced when water content is increased, also at saturation the resilient modulus can be reduced to one third compared to the value at low water content.

#### Density:

Density of aggregates related to how the single grains interact with each other, can be considered one of the most important factors influencing the stiffness and resistance to permanent deformation. Many studies have been performed in order to understand and evaluate the effect of density to other parameters. For instance cyclic load triaxial tests were performed in order to study the effect of density on the deformation behavior of granular packing (Barksdale, 1972). He observed from experimental data that, generally, an increase in compaction energy, expressed as percent of maximum Proctor dry density, increases stiffness and resistance to permanent deformation.

The effect of density on the resilient modulus was also studied by Hicks and Monismith they observed that the effect of density on the resilient modulus was greater for a partially crushed material than for a crushed material and that the effect of density decreased with increasing fines content (Hicks and Monismith, 1971).

Another aspect that was investigated related to the degree of compaction and grading. Van Niekerk conducted experiments where he was able to conclude that the resilient modulus in general increases significantly with increasing the degree of compaction (Van Niekerk, 2002). Also rate of the increase was found to be related to the grading.

Dry density is a key parameter for the material, well-graded material up to a certain level of fines content is mostly influenced by the dry density (Uthus, 2007). Grading with a relatively high amount of fines seems to be important under dry conditions, while density seem to override the effect of the degree of saturation for gradation with  $n=0.35$ .

Unbound granular layers in pavements are subjected to many parameters that influence their behavior. Many parameters have been stated to better understand the complex mechanical behavior to which the granular material respond depending on its stress history and the current stress state in addition to the degree of saturation and density.

## **2.5 Hydraulic Bound Layers**

Hydraulically bound layers are obtained by mixing aggregates or granular soils with cement (or other hydraulic road binders) with a certain amount of water, to produce a material whose strength is greater than that of the original unbound material. Hydraulically bound mixtures have been used in road, airfield, port and other pavement construction for over 50 years.

One of the most used mixing stabilizers throughout the world is Portland cement. The chemical reaction that takes place between cement and the aggregates, in the presence of moisture, produces hydrated calcium aluminate and silicate gels, which crystallize and bond the material particles together. In general, the strength of the material will gradually increase with a rise in the cement content. Materials treated by cement are described variously as soil cement, cement-treated materials, or cement-stabilized materials. If granular aggregates are used (crushed rock or natural gravel) these are called cement bound granular material.

Cement can be normally used to stabilize any type of soil, except soils with organic content greater than 2% or having pH lower than 5.3 (ACI Committee, 1997). Normal range of cement percentage requirement (by soil weight) for A-1-A soils is 3%-5%, and for A-6 soils is 9%-15%.

Studies conducted by Kezdi reported that cement treatment slightly increases the maximum dry density of sand and highly plastic clays but it decreases the maximum dry density of silt (Kezdi, 1979). Significant effects of cement-soil stabilization is reduction in shrinkage and swell potential, increase in strength, elastic modulus, and resistance against the effect of moisture, freeze, and thaw.

Also granular aggregates are mixed with cement, as we know granular aggregates are the primary road building material used in road construction and are a non-renewable natural resource. For this reason to limit the exploitation of non-renewable natural resources studies have been conducted on the possibility of using marginal granular materials as a valid alternative in cement bound granular material. Such materials have normally high fines and high fine sand content that can predict they use. The possibility of using cement stabilization on this type of aggregates has been considered and studied with satisfactory result (Berthelot et al., 2010).

## 2.6 Bitumen Bound Layers

### 2.6.1 Theories and Mechanisms of Adhesion

The use of bitumen as a binder to glue together aggregates contributing to the creation of asphalt pavement layers can go back as far as the late 1800. The binder aggregate bond can be subjected to major or minor stresses to which it must resist in order to ensure the durability and integrity of the roadway. The key factor that had to be considered was adhesion; it seems that this topic of research between aggregate and bitumen started to be considered in the early 1920. One of the main concerns was adhesive failure induced by water entering the bituminous mix, called moisture damage, or simply stripping. In the early 1930s in order to resolve adhesion problems in bituminous mixtures ‘The Committee on Interfacial Surface Tension’ was established. The most significant result of research during this time was identification of chemical compounds that could be used as adhesion promoters between bitumen and aggregate.

Adhesion is a very complex phenomenon that involves a multidisciplinary science, for this reason the study of adhesion uses various concepts. Many theoretical models have been proposed and will be discussed shortly.

- 1) Mechanical interlocking
- 2) Electronic theory
- 3) Theory of boundary layers and interphases
- 4) Adsorption (thermodynamic) theory
- 5) Diffusion theory
- 6) Chemical bonding theory

#### **The mechanical interlocking model:**

This theory can be considered one of the most intuitive adhesion phenomenon’s. The mechanical interlocking was proposed by Mac Bain and Hopkins in 1925 (Mac Bain and Hopkins, 1925) considered that there were two kinds of adhesion, specific and mechanical. Specific adhesion involved interaction between the surface and the adhesive: it could be chemical or absorption. Mechanical adhesion was only considered possible with porous materials. Despite its “obvious” nature, the mechanical theory of adhesion fell out of favour, and was largely rejected by the 1950s and 1960s. However, by the 1970s the mechanical theory was again being taken seriously.

Pocius (Pocius, 1997) improved adhesion through mechanical effects with the following theories:

- Physical “lock and key”: The lock and key effect when the adhesive phase penetrates in the pores of the solid substrate resulting in a physical anchoring between the two materials.
- Redistribution of stresses: stresses are transferred across or into the adhesive, which in most cases exhibits viscoelastic and plastic flow properties.
- Increased surface area: surface roughness improves adhesion purely due to an increase in physical area of contact.

It is possible to state that aggregates with a porous, slightly rough surface will promote adhesion by providing the elements for a mechanical interlocking effect.

However, the surface texture of an aggregate also affects its capacity of being coated (coatability), or wettability, in that a smoother surface coats easier than a rough surface (Tarrar and Wagh, 1992). Criticism to this theory state that, as suggested from different studies, improved adhesion doesn't necessary results from a mechanical interlocking.

#### **The Electronic theory:**

The introduction of this theory was primarily introduced by Deryaguin in 1948. It is known that solid surfaces can be characterized as electropositive or electronegative. Essentially the electropositive material donates charge to the electronegative material thereby creating an electric double layer at the interface (Deryaguin and Smilga, 1969).

According to this it is possible that an electron transfer mechanism between the substrate and the adhesive, having different electronic band structures, can occur. No clear correlation between electronic interfacial parameters and work of adhesion is usually found. It could be concluded that the electrical phenomena often observed during failure processes are the consequence rather than the cause of high bond strength.

#### **Theory of boundary layers and interphases:**

This theory was introduced to understand the alterations and modifications of the adhesive and/or adherent that can be found in the vicinity of the interface. Bikerman introduced a first approach to this problem he stated that cohesive strength of weak boundary layer (WBL) can always be considered as the main factor in determining the level of adhesion, even when the failure appears interfacial (Bikerman, 1961). According to this assumption, the adhesion energy “G” is always equal to the cohesive energy G of the weaker interfacial

layer. Many physical and chemical phenomena are responsible for the formation of such interphases and can be summarised as follows:

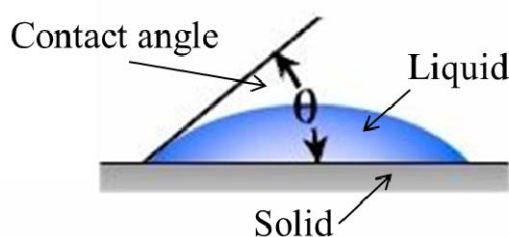
- 1 the orientation of chemical groups
- 2 migrations toward the interface of additives or low-molecular-weight fraction
- 3 the growth of a trans-crystalline structure
- 4 formation of a pseudoglassy zone resulting from a reduction in chain mobility through strong interactions with the substrate

**Adsorption (thermodynamic) theory:**

This theory named adsorption or thermodynamic theory is generally attributed to Sharpe and Schonhorn and is the most widely used approach when studying adhesion at present (Sharpe and Schonhorn, 1963).

Thermodynamic theory is based on the concept that an adhesive will adhere to a substrate due to established interatomic and intermolecular forces at the interface provided that a contact is achieved. The forces result from Van der Waals and Lewis acid-base interactions. The formation of contact step through a liquid-solid is related to the magnitude of the force that can generally be related to fundamental thermodynamic quantities. Good adhesion becomes generally an essential criteria of good wetting, although this condition is necessary but not sufficient. The terms such as wetting, spreading and contact angle have become of common use when dealing with adhesion.

The shape of a liquid drop on a surface can be described by the contact angle ( $\theta$ ) between them Figure 3.



**Figure 3. Contact angle between liquid and solid**

Wetting of a surface is the process where the adhesive comes into contact with the surface and establishes fundamental forces of adhesion. For complete spreading, the contact angle equals zero and the adhesive spreads spontaneously over the surface (Myers, 1991). Viscosity of the liquid is also another parameter that influences wetting and spreading, but also parameters like roughness and heterogeneity of the solid surface are important key factors. From a thermodynamic point of view, wetting and spreading depends on the competition between adhesive forces and cohesive forces.

#### **The diffusion theory:**

The diffusion theory attributes the adhesion on the assumption that the adhesion strength of Polymers to themselves (autohesion) or to each other are due to mutual diffusion (inter-diffusion) of macromolecules across the interface. This theory was supported by Voyutskii (Voyutskii, 1963). He studied the adhesion of polymers to each other considering the duration of contact between adhesive and substrate, temperature, molecular weight of adhesive, molecular shape and polarity. This is of great importance for many adhesion problems, such as healing and welding processes.

#### **Chemical bonding theory:**

This theory considers the formation of chemical bonds across the adhesive-substrate interface and these can participate to the level of adhesion between both materials. These types of bonds can be considered primary bonds but also there are secondary bonds that consider physical interactions (Schultz and Nardin, 2003). The formation of chemical bonds it clearly depends on the reactivity of both adhesive and substrate. Primary and secondary bonds are terms also used in order to state the strength or bond energy.

The interaction chemistry of bitumen-aggregate is complex and variable due to the complex and variable composition of the materials involved (Petersen et al., 1982).

One field of interest involving interfacial chemical bonds is the use of adhesion promoters' molecules that act as a bridge improving joint strength between adhesive and substrate. These adhesion promoters react chemically on both ends with the substrate on the one side and the polymer on the other.



In conclusion it is possible to say that adhesion is a very complex phenomenon to study, that involves several mechanisms that can act simultaneously. Depending on the variety of material to be bonded and conditions, the need of several theories are needed in order to explain all the mechanism involved. Considering the bitumen-aggregate system, adhesion is explained by the theories that were previously introduced: boundary layers, mechanical theory, electrostatic theory, chemical bonding theory, and thermodynamic theory.

However, it is generally assumed that the adsorption or thermodynamic theory defines the main mechanism exhibiting the widest applicability. It describes the contact and the development of physical forces at the interface that is a necessary step for: interlocking, inter-diffusion and chemical bonding.

The importance of physical characteristics of the aggregate is introduced in these theories. Although surface morphology can promote adhesion, it can also have the potential to cause weak boundary layers if selective absorption occurs. Wetting of the aggregate is another important prerequisite for good adhesion and does not only rely on good mixing conditions, but also on the characteristics of the aggregate surface.

## **2.7 Digital Image Processing**

Digital image analysis and computer vision have grown rapidly in the past decades. The use of digital images as a means for representing and communicating information is becoming of common use in several important areas for example medical imaging, material science, biology and in many fields of engineering. The introduction of Digital Image Processing (DIP) technique is a powerful tool when morphological information are required to better understand a behavior of a material, sort out objective determine physical parameters. These types of characteristics are often studied in civil engineering, when analyzing materials behavior and characteristic. The use of image analysis is becoming a more common tool in the field of civil engineering.

DIP technique like other image analysis consists of a number of operations performed in sequences to obtain digitalized pixel images in order to extract information's. It has the major advantage that in some cases can be almost automatic, fast and easy to perform. The basic operations include image acquisition, processing, measurements, data processing and interpretation (Bons and Jessel, 1996). During the 1990s the use of DIP in order to study size and shape of aggregates was attempted by Barksdale (Barksdale et al., 1991), Li (Li et al., 1993), Yue (Yue and Morin, 1996), useful results were obtained. One of the major problems associated with DIP to measure particle size and shape is that works with only two dimensional projections. Consequently, the third dimension that is the thickness of the particles can't be obtainable directly from the DIP results. Also in order to perform correct measurements the particles must be carefully spread out to avoid touching or overlapping. To avoid inaccurate measurements the particles must not appear too small in the pixel representation. With the introduction of new techniques and machines and also the development of software's these limitations are starting to be reduced. The introductions of techniques that are able to capture images from many different directions are a powerful tool in order to better characterize the geometry of the aggregate.

Another useful tool that is based on image analysis to perform measurements on aggregates is called Aggregate Imaging System (AIMS) developed at Texas Transportation Institute.

This method consists of a computer-automated video system that directly analyzes texture, angularity and shape of aggregates (Jagan et al., 2006), with aggregate dimension ranging from 37.5 mm to 150 mm (Masad, 2004). A unique feature of this system is the ability to capture texture through wavelet analysis of black and white images.

Other applications of Image analysis have been studied like the possibility of using this technique to be a valid alternative to determine particle size distribution instead of performing mechanical sieving.

Attempts have been made on determine aggregates gradation in both asphalt and concrete mixtures; the difficulty is due on distinguishing boundaries between adjacent, colourless grains, textural features and twin boundaries (Fueten, 1997).

Image analysis was performed for detecting aggregate gradation in asphalt mixture from planar images (Bruno et al., 2011). The purpose of the study was finalized on detecting an effective method to analysis asphalt section images to extract aggregate gradation automatically without the need of separation of the bitumen from the aggregate.

Image analysis was also used to study aggregate orientation in asphalt mixtures subjected to different methods of compaction (Hunter et al., 2004). This in order to evaluate if different compaction methods: gyratory compaction, vibratory compaction and slab compaction, will influence orientation and distribution of the aggregates and relating this to mechanical performances.

The study of thin sections was also performed using image analysis to investigate mortar morphology in particular obtain the binder/aggregate ratio and the grading curve instead of performing mechanical sieving method (Marinoni et al., 2004).

Image analysis is becoming more widely used in civil engineering studies, with many possible applications especially in pavement engineering. The aim of the reported studies have one characteristic in common they all want to obtain information's in a more automated and fast way replacing traditional mechanical methods to perform the same measurement.

### **3. METHODOLOGY AND PROCEDURES**

#### **3.1 Introduction**

This chapter introduces the research methodology used for this study and how it has guided tests and data analysis. When dealing with materials that can be considered a waste the main concern is to consider regulations and legislations that deal with this matter. For this reason all the procedures imposed by regulations must be strictly followed. To do so tests must be performed according to Italian specifications. Once tests have been conducted and it is possible to state that the material that we are dealing with is not a waste and doesn't releases hazardous substances it is possible to characterize the material for its final use.

Most of the studies that were found in literature about the use of granite by-products focused the attention on using granite by-products fines to be used as fillers in HMA layers, while the use and behaviour of granite by-products in other road layers was not always taken in to consideration.

In this research the possible application of granite by-products for all pavement layers is taken into consideration. Tests were initially performed to use granite by-products to be used as aggregate for unbound granular pavement layers. But also to evaluate the possible use in hydraulically bon layers, using Portland cement as road binder.

This research focused major attention on using image analysis, to better understand the parameters that influence adhesion behaviour between granite by-products when mixed with bitumen.

#### **3.2 Selection of the Case Study**

In order to verify the potential use of Sardinian granite by-products for road pavement layers and also verifies if different processes to which granite is subjected can influence the performance. For this reason granite deriving from the same mother rock but subjected to different excavation techniques was needed. The problem was of not simple solution finding granite of the same type but subjected to different excavation techniques in the same construction site or same quarry was difficult to find. A constructing site was found on the south east coast of Sardinia consisting on the excavation of tunnels for the realization of the new road S.S.125.

The project consisted on the excavation of three main tunnels called: Marapintau (1.290,00 m), Istellas (212,50 m) and Murtineddu (2.590,50 m) with a total length of 4.1 km, also two service tunnels were constructed for the Marapintau and Murtineddu galleries these had nearly the same length of the main galleries. The tunnels were being excavated on compact and homogeneous granite bodies with a total of 800.000 m<sup>3</sup> of extracted material. Two techniques were used on this site: TBM for the service tunnels and D&B for the galleries, also the larger blocks of granite by-products obtained from D&B were reduced in size with a mobile crusher machine. At this point three types of granite by-products could be tested each deriving from the same mother rock but subjected to different processes. The three materials were divided in different stock piles with the possibility of conducting periodic sampling campaigns according with the progress of the works in order to have an overall picture of the type of granite by-products we were dealing with.

Useful informations were obtained considering other constructing sites dealing with granite and using different excavation techniques, like the new Gotthard tunnel project (Pepino, 2009) in which special attention is being given to the millions of tons of granite that are being excavated.

### **3.3 Preliminary Characterization**

Samples were collected from different stock piles and once in the laboratory they were reduced in order to conduct physical and chemical characterization in order to evaluate the exact type of granite we were dealing with. This was important to establish from a geological point of view the area in which the tunnels were being excavated and to better understand Sardinian granitoids distribution. In this phase specific tests were conducted according to Italian regulations that deal with excavated earth and rocks. While the position of the construction site didn't indicate recent or past of pollution activities, that could have contaminated the area subjected to excavation. In fact the site was located in an area where no anthropic activities had never taken place like industrial or civil. Special attention was given considering hazardous elements that could have contaminated the material during the various processing phases of the construction site.

### **3.4 Characterization of Granite By-Products for the Correct Use in Road Pavement Layers**

Each of the tests conducted to use granite by-products in both unbound and bound layers were performed according to Italian regulations considering CNR (Italian National Center for Research) and UNI-EN (Italian Organization for Standardization) specifications. This because in Italy in many cases the two technical specification are used depending on the field of application and on the tests requested, with the limitation that CNR specification are only valid in Italy. In some cases tests were performed not only following regulations but also verifying the possibility of introducing new procedures to evaluate and study parameters. The results obtained performing the tests on granite by-products were compared not only with the limits imposed by the specifications but also with values that normally are achieved on aggregates currently used in road pavement layers. Also to understand if different processes to which granite was subjected could modify characteristics and behaviour, the results obtained from the three tested materials were compared. For the unbound and hydraulic bound layers the approach was that of following Italian specifications.

For bound bitumen layers, due to the known difficulties of adhesion between granite and bitumen, image analysis technique was introduced to better understand the parameters that influence such phenomena.

### **3.5 The Use of Image Analysis**

The use of static image analysis was introduced while performing tests to evaluate the affinity between aggregates and bitumen. It is known that adhesion at the interface between bitumen and aggregate is one of the principal functional properties to guarantee durability of asphalt mixes. The presence of water can reduce adhesion between bitumen and the aggregate surface, such phenomena is known as stripping (Daučík et al., 2011). Susceptibility to stripping is an indirect measure of the power of a binder to adhere to various aggregates, or of various binders to adhere to a given aggregate. The stripping method tests consist on evaluating the bitumen layer on the aggregate surface, by visual observation, after that the coated aggregate have been subjected to straining conditions.

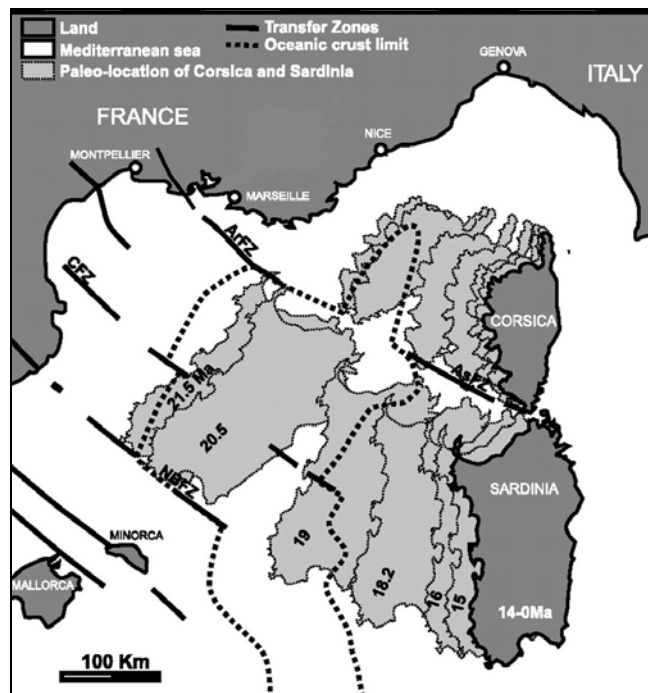
In order to have a more scientific and precise method to measure the percentage of surface of the aggregates coated by bitumen the possibility of taking pictures and to examine them using an image analysis software was considered. Several image analysis softwares were analyzed and two of them were chosen to be calibrated.

## 4. SARDINIAN GRANITOIDS ROCKS AND THEIR EXPLOTATION

### 4.1 Introduction to the Geology of Sardinia

The following chapter will introduce informations on the geology of Sardinia focusing the attention on granitoids rocks. Also the geological composition of the Sàrrabus-Gerrei region will be introduced.

Sardinia, with its 24.089 km<sup>2</sup> is the second largest island in the Mediterranean after Sicily. The present position of Sardinia is due to a 30° anticlockwise rotation of the Corsica-Sardinia block away from Europe caused by opening of the Western Mediterranean Ligurian-Provençal basin (Carosi et al., 2006). The age of the rifting phase is dated to Oligocene followed by a short early Miocene oceanic accretion. Steps of anticlockwise rotation Corsica-Sardinia block are reproduced in Figure 4 (Gattacceca et al., 2007).

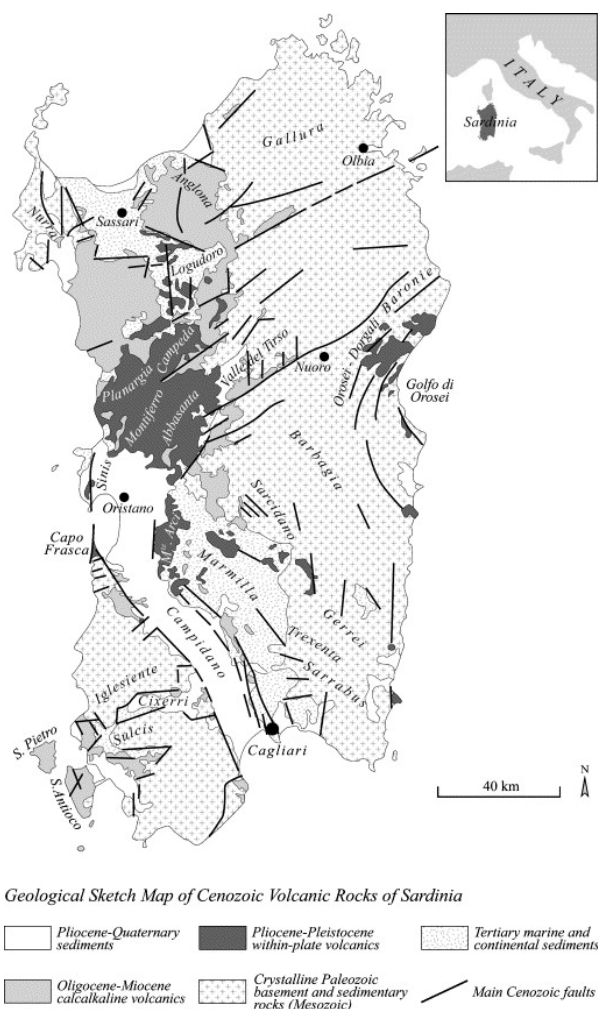


**Figure 4. Geodynamic context of the study area in the frame of Corsica-Sardinia anticlockwise block rotation and Liguro-Provençal basin opening**



In Sardinia sedimentary, magmatic and metamorphic records are well represented within three main lithological complexes Figure 5:

1. Paleozoic basement that underwent repeated phases of deformation and metamorphism during the Caledonian and Hercynian orogenic cycles, and was eventually intruded extensively by calc-alkaline granitoids
2. Late Palaeozoic epicontinental sequence and a Mesozoic carbonate platform sequence, representative of stable shelves, that formed the passive margin of Southern Europe
3. Cenozoic to Quaternary cover consisting of shallow-water marine carbonates, siliciclastic sediments, continental conglomerates, as well as volcanic rocks represented by a calc-alkaline suite and alkaline basalts



**Figure 5. Geological sketch map of Sardinia**

## **4.2 Sardinian Granitoids Rocks**

Granite is an igneous intrusive acid rock with a granular structure. Igneous rocks formed when molten rock (magma) cooled and solidified, with or without crystallization, either below the surface as intrusive (plutonic) rocks or on the surface as extrusive (volcanic) rocks. The term granite groups a number of different rocks whose scientific name are tonalite, granodiorite, monzogranite, sienyte, and other rocks containing varying amounts of quartz. All these varieties are more properly named as granitoid rocks.

The informations acquired on Sardinia granitoids are still incomplete because large portions of the batholith still require field mapping. A batholith is a large emplacement of igneous intrusive rock that forms from cooled magma deep in the Earth's crust. Although they may appear uniform, batholiths are in fact structures with complex histories and compositions.

The granitoids forming the Sardinian batholith have extremely composite characteristics and are represented from different petrographic terms: monzogranite, leucogranite, granodiorite and tonalite.

The syntectonic granites represent less than 1 % of the total area occupied by the granite in Sardinia, they are characterized by oriented textures in the arrangement of minerals constituents have predominantly tonalitic-granodiorite composition and are especially in Gallura and Goceano Figure 6.

The late-tectonic granites make up about 74% of Sardinian batholith and are composed of tonalite granodioritiche, granodiorite and monzograniti. In particular monzogranites and granodiorites occupy about 65% of the intrusive structure of the Sardinian island. This granitoids have colors that varies from gray to pink (if characterized by large crystals of pink orthoclase) and are extremely common in the following regions: Gallura, in some areas of Ala dei Sardi and Buddusò at Benetutti in Nuoro, Ogliastra and southern Sarrabus Figure 6. The post-tectonic granites constitute 25% of the batholith and are represented by leucogranitie, they form a series of large plutons limited in some areas of Gallura, in the area of Ala dei Sardi, northern Sarrabus and Sulcis-Iglesiente Figure 6.



**Figure 6. Different regions of Sardinia**

The following geological picture of Sardinian granitoids is based mainly on surveys and studies conducted by Ghezzo (Ghezzo et al., 1972) and Di Simplicio (Simplicio et al., 1974) and have been recently integrated with studies on selected but limited areas of the batholith. The post-collisional Hercynian batholith of Sardinia results from the union of many calc-alkaline plutons of prevailing monzogranitic to leucogranitic composition, with subordinate granodiorite and tonalite and occasional gabbroic rocks (Ghezzo et al., 1972) and (Bralia et al., 1982).

The genesis of the Sardinian granitoids is still a matter of debate, and no single and generally accepted model exists. A mixing model of mafic and acid magmas is generally assumed to explain the textural, petrochemical and isotopic features of the tonalitic/granodioritic rocks (Bralia et al., 1982).

Although in Sardinia pre-hercynian magmatism and deformations were recognized, the structure of the Island is essentially related to the hercynian cycle (Secchi and Lorrai, 2001).



**Figure 7. Simplified geological sketch map of late-hercynian granitoids of Sardinia**  
(source Secchi and Lorrai, 2001)

**Legend:**

- (1) undifferentiated post-Palaeozoic sedimentary and volcanic covers;
- (2) late Palaeozoic volcanic and sedimentary covers;
- 3-5: late-hercynian granitoids.
- (3) meta (a) and peraluminous (b) leucogranites;
- (4) monzogranitic granodiorites and monzogranites;
- (5) granodiorites;
- 6-8: metamorphic basement;
- (6) low-grade metasedimentary and metavolcanic sequences of more external zone;
- (7) low to medium grade metasedimentary and metavolcanic sequences of «nappe zone»;
- (8) high grade metamorphic complex (paragneisses, micaschist and quartzites) with migmatites of axial zone;
- Other symbols: (9) main over thrusts; (10) Posada-Asinara line; (11) main regional faults



**Legend:**

- (1): Quaternary sedimentary deposits, Plio-Quaternary volcanics (Capo Ferrato area) and Oligo-Miocene sedimentary covers (Ussana Fm). 2-6: Late-Hercynian granitoids of Sàrrabus pluton.  
(2): Fayalitebearing biotite granites (Sette Fratelli type granite);  
(3): biotite leucogranites (S. Priamo type leucogranites);  
(4): monzogranitic granodiorites and monzogranites (Monte Nai type monzogranite);  
(5): hornblende biotite granodiorites (a) with gabbrotonalitic masses (b) (Cala Regina type granodiorite);  
(6): two-pyroxene gabbrotonalites of Burcèi area (Burcèi type gabbrotonalite);  
(7): Palaeozoic metamorphic Basement;  
(8): main faults. Note that in order to simplify late-hercynian dike swarm was not plotted.

**Short description of the main complexes:**

1. Burcèi gabbrotonalitic body: Geological and petrographical features of this body have been outlined by Brotzu (Brotzu et al., 1993). Schematically, it is a composite sill-like body located in the northernmost part of the Sàrrabus pluton, at the contact with Palaeozoic basement. It is constituted of two separate injections of magma ranging in composition from two-pyroxene biotite gabbrotonalites to orthopyroxene-bearing granodiorites.
2. Granodiorites: Granodiorites constitute a number of discrete intrusions separate by sharp contacts widespread in the southern side of Sàrrabus pluton. The granodiorites outcropping in the southernmost area are enriched in dark enclaves and display a moderate magmatic foliation. These rocks are equigranular medium-grained rocks and, under the microscope, they are better defined as hornblende biotite granodiorites, while the granodiorites occurring in the northernmost portions are essentially biotite granodiorites straddling to the boundary with the monzogranites.
3. Monzogranites and leucogranites: Monzogranites outcrop in the central part of the pluton. These rocks are coarsegrained, light grey, biotite rocks. Dark enclaves are rare. These rocks are quite often crosscut by sub-vertical aplogranitic dikes.
4. Leucogranites: Leucogranites outcrop widely in the northern side of the pluton. These rocks are coarse-grained, pinkish, biotite rocks. Dark enclaves are very rare, while biotite-bearing pegmatitic pockets are quite commonly observed. These rocks are quite often crosscut by sub-vertical aplogranitic dikes; mafic dikes are quite rare.
5. Fayalite-bearing biotite amphibole monzogranite: Light greysh leucocratic medium to coarse-grained rocks. Dark enclaves are quite rare. These granites are characterized by the common occurrence of Fe-hastingsitic amphibole and Fe-

biotite as mafic phases, as well as fluorite in accessory amounts. Some typical pseudomorphs suggest the previous existence of subordinate fayalite. The SF granites are located in the central part of the Sàrrabus igneous massif; they crosscut with vertical contacts the surrounding monzogranites.

#### **4.4 Exploitation of Sardinian Granitoids**

In Sardinia quarrying and granite working started from the Nuragic culture and developed during the Roman age and has grown becoming an important economical resource for the region. Since the commercialization of Sardinia granite has become an organized trade (1870) there are four main ornamental quarrying districts: Arzachena-Luogosanto, Tempio-Calangianus, Buddusò-Ala dei Sardi and Ovodda, such districts are located for the majority on the north east of Sardinia. Short description of the lithology that characterize Sardinian granite quarrying districts will follow:

1. **Arzachena-Luogosanto** characterized by heterogranular monzogranite with small pink K-feldspar crystals (1-2 cm);
2. **Tempio-Calangianus** characterized by heterogranular monzogranite with large pink K-feldspar crystals (8-10 cm );
3. **Buddusò-Ala dei Sardi** characterized by equigranular leucomonzogranite with medium-small K-feldspar crystals (1-3 cm), plagioclases and light gray quartz;
4. **Ovodda** characterized by heterogranular monzogranite with small K-feldspar crystals (less than 2 cm ), grey plagioclases and quartz;

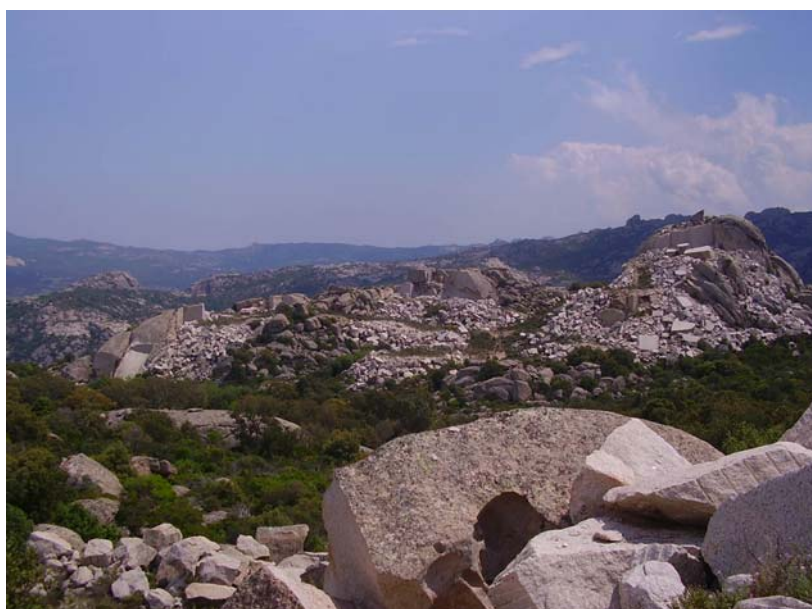
Each district is always characterized by good geological homogeneity, although sometimes there are little extended lithologies different to the ones that characterize the quarry district but equally subjected to cultivation. Due to a drastic increase in the market of Chinese granite many of the quarries of the Sardinian districts are no longer in production and material lies abandoned see Figure 9.





**Figure 9. Abounded Granite quarry based in the locality of Monte Cilata district of Luogosanto**

Large areas of the cultivation sites are covered by abounded stockpiles in which are possible to find from large shapeless blocks to granular materials. Examples of what remains after the exploitation of the ornamental stone quarry industry are shown in Figure 10 and Figure 11.



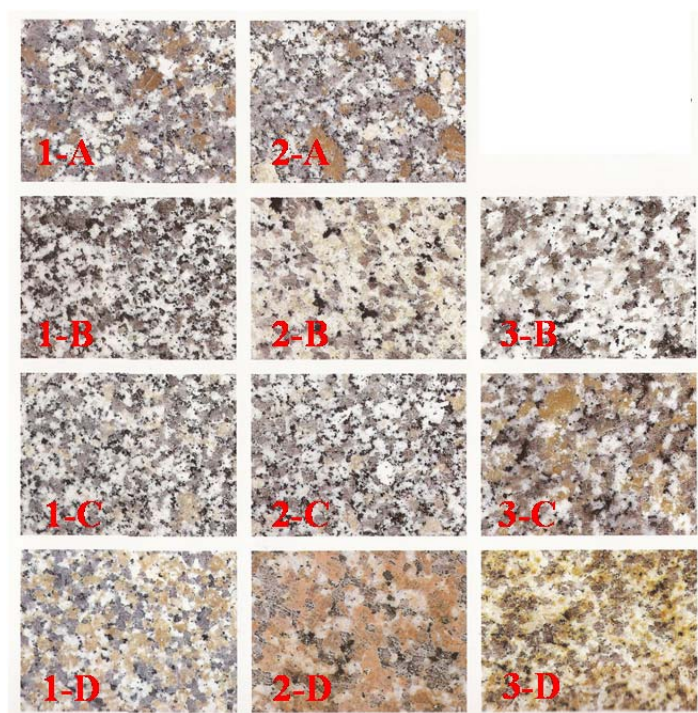
**Figure 10. Abounded Granite quarry based in the locality of Monte Muddetru district of Calangianus**





**Figure 11. Abounded Granite quarry based in the locality of Stazzo Carrulu district of Calangianus**

In total there are 11 types of commercialized granite in Sardinia (see Figure 12) that differ not only in colour but in some cases for physical and mechanical properties: **ghiandone gallura (1-A)**, **giandone limbara (2-A)**, **malaga gray (1-B)**, **nuraghe gray (2-B)**, **pearl gray (3-B)**, **antique pink (1-C)**, **beta pink (2-C)**, **cinzia pink (3-C)**, **nule pink (1-D)**, **San ferula pink (2-D)** and **Giacomo yellow (3-D)**; these are all the types of commercialized granite that can be found in Sardinia.



**Figure 12. Commercial granite types**

The family of granodiorites and monzogranitic granodiorites are related to the commercial granite named **malaga grey**, this is mainly present in the area of Buddusò-Ala dei Sardi (north-central Sardinia) and in the area of Villasimius (South east coast).

Leucogranite correspond to the family of the commercial types named: **cinzia pink**, **nule pink** and **ferula pink**, characterized by structures that can be both equigranular and inequigranular with grain size ranging from medium to large crystals of K-feldspar.

Leucogranite also belong to the commercial type named **San Giacomo yellow**, with medium-grained and K-feldspar.

To the pinkish monzogranite with trends ranging to equigranular with pinkish K-feldspar with size ranging of no more than two centimetres, match the commercial type named **beta pink**. Quarries that commercialize this type of granite are located in the district of Arzachena Luogosanto and Tempio, forming 41% of the active quarries that extract monzogranite.

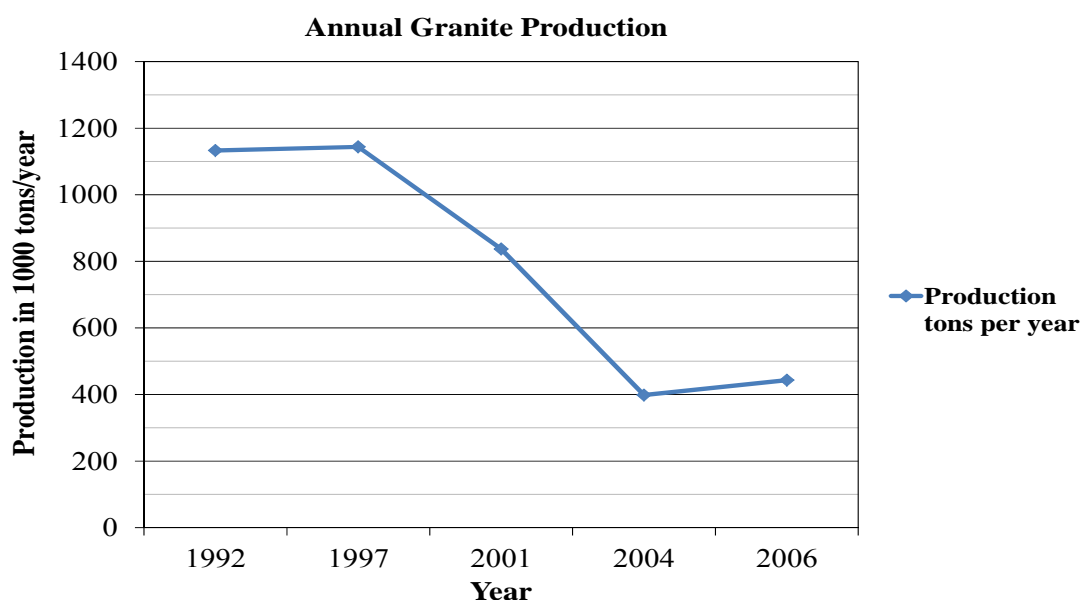
The commercial granite named **ghiaudone** divided in **ghiaudone limbara** and **ghiaudone gallura** corresponds to a pink monzogranite with texture moderately oriented characterized by the large size of some individuals K-feldspar which can sometimes reach up to 8-12 cm in size.

Medium-grained monzogranite with a uniform texture and with plagioclase and little biotite correspond the commercial granites named **pearl gray** and **Sardo gray**.

The commercial type named **nuraghe gray** belongs to the class of monzogranite with medium-grained granodiorite monzogranitiche moderately disequigranular, K-feldspar euedrali small with size ranging from 1 to 2 cm, colour pearly-white or pale pink and a percentage of biotite greater than the **Sardo gray**.

The inclusion of some commercial types in different petrographic families depends on the fact that, in the absence of a specific law, the assignment of names to the lithological types is done by the owners of the quarries without following any precise directive.

Quarrying of materials for ornamental use is characterized by a strong territorial concentration. As already said the main Sardinian granite quarry activity is located in two major areas: Gallura and Goceano Figure 6. According to the Department of Industry of the autonomous region of Sardinia, from data collected in 2006 on the bases of the informations given by the owners of the quarries the annual production of granite has been of 443.000 tons. From the data collected considering the annual production from 1992 to 2006 it is possible to see that the annual production has been decreasing drastically passing from nearly 1.2 million tons between the years 1992-1997 to just over 400.000 tons in 2006 Figure 13.



**Figure 13. Annual granite production (source Department of Industry of the autonomous region of Sardinia)**

This reduction of granite extraction has caused inevitably to the closure of many quarries passing from n°182 active quarries in 1992 to n°126 in 2006. Granite quarries produce approximately 330.000 tons per year of granite wastes. This material lies abounded near the area of excavation or transported to nearby pits. For this reason a new life must be given to this type of material considering it no longer a waste but a new commercial product to be used in the construction industry.

## **5. CASE STUDY GRANITE BY-PRODUCTS FROM TUNNELING WORKS**

As widely introduced in the previous chapter in Sardinia large portions of territory are occupied by stockpiles formed of abandoned granite by-products. This type of material derives mainly from the past and present quarry industry but also from civil engineering works that often have to deal with such rocks due to the geology of Sardinia. The three major types of granite rocks that can be found in Sardinia are Monzogranite, Leucogranite and Granodiorite, of these Monzogranite is the more widely present, for this reason many of the abandoned stockpiles are composed of such material. The composition of the stockpiles in terms of size and shape depends on the process to which the material has undergone and can be divided as follows: third choice blocks, large shapeless blocks, small shapeless blocks and also small size particles like sand or fines.

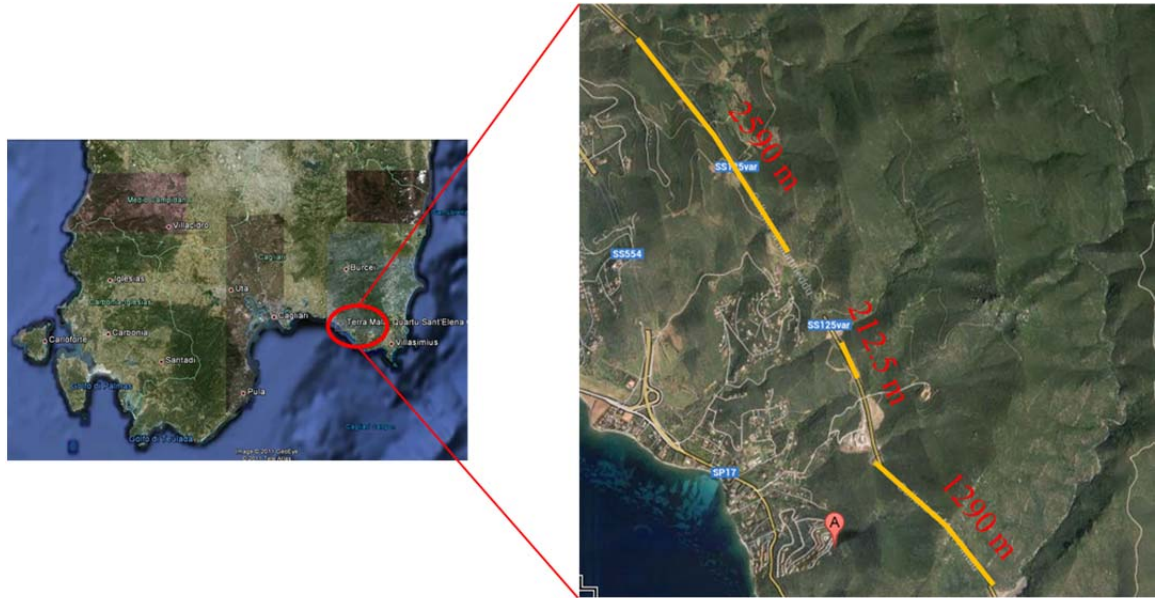
In this research the possible applications of granite by-products to be used in road pavement layers was analyzed. The research approach was that of focusing the attention on a case study in which only one type of granite could be considered but subjected to different types of processes. The choice was made by considering a road construction site in which 5 tunnels were being excavated in compacted and homogenous granite. The construction site was located in the Sarrabus-Gerrei region (see Figure 14), this area can be located on the south east coast of the island, in which from a geological point of view the types of granite that can be found are mainly granodiorite and in some cases monzogranite.



**Figure 14. In red the position of the area involved by the construction site**

The site in question was part of major road works for the construction of the new S.S.125. In this site the aggregates deriving from different excavation processes and apparently belonging to only one single type of granite could have been stated. With the possibility of verifying if different processes to which granite is subjected can influence not only mechanical characteristics but also the behavior depending on the layer in which this material is used.

The construction project consisted of the excavation of three main tunnels called Marapintau (1.290,00 m), Istellas (212,50 m) and Murtineddu (2.590,50 m) (see Figure 15), of which Marapintau and Murtineddu were provided with service tunnels, with an estimated excavated material of 800.000 m<sup>3</sup>.



**Figure 15. Satellite views of the south of Sardinia and of the area involved by the construction of the tunnels highlighting in red the length**

The excavation techniques used in this project were of two types Tunnel Boring Machine (TBM) for the service tunnels and Drilling and Blasting (D&B) for the main tunnels ( Figure 16). Also to reduce the large blocks of granite deriving from D&B a mobile jaw crusher was used with a feed size ranging from 600-900 mm. Considering the two different types of excavation techniques and the material deriving from the mobile crusher in this site three potential types of granite by-products were available. Each of them was divided in different stockpiles in easy and accessible areas, this was important in order to sample the material in a correct manner. The maximum daily advance performed using D&B method was of 5 m but in some cases it could drop to 0.5 m this due to the mechanical characteristics of the rock. While the average daily advance for the TBM was of 30 m, but at times peaks of 45 m were reached.





**Figure 16. Entrance of one of the main galleries and service tunnels**

The TBM that was used was an open shield one with a 4 m diameter, manufactured by Aker Wirth. It is possible to see the TBM without the cutting head in Figure 17 this picture was taken once the TBM had finished the excavation of the last of the two services tunnels.



**Figure 17. TBM without the cutting head**

The D&B excavation method used for the main tunnels consisted on using jumbo drills machines (Figure 18) equipped with a hydraulic drilling system, in order to drill the holes forming a correct pattern in which place the explosive. The explosive that was used was of slurry and gelatin type with charges of 1 kg for each m<sup>3</sup> of rock.

These types of explosives have an efficiency of 64% depending on the type of rock that they have to drill with.





**Figure 18. Jumbo drill in operation, source (<http://www.todini.it>)**

The mobile jaw crusher was an “Om Track Ulisse tk096f” type (Figure 19); this machine could reach a production of 190 tons per hour. This depending on the application and the type of material that had to deal with.



**Figure 19. Mobile crusher in operation**

The granite deriving from the excavation processes could be separated in three different types. Material deriving from TBM (see Figure 20) homogenous in terms of size and shape this due to the way in which the cutters are able to penetrate in to the rock producing fragments with a flat and elongated shape.

This is not possible to have with materials deriving from D&B in this case the material is not homogenous, with sizes ranging from large blocks to sand or gravel. The material deriving from D&B is mainly the one that can be found in many of the old abounded Sardinian granite quarries, this because in the early days before diamond wire saws were introduced, blasting technique was one of the most used.



**Figure 20. Stockpile of granite deriving from TBM excavation**

In Figure 21 it possible to see material deriving from D&B, due the heterogeneity of the size and shape, the stockpile was once again reduced, by collecting the larger blocks in order to be reduced by the crusher.



**Figure 21. Stockpile of granite deriving from D&B excavation**

## **6. LABORATORY TESTS ON GRANITE BY-PRODUCTS TO BE USED IN ROAD PAVEMENT LAYERS**

### **6.1 Material Used in Test Campaign**

In order to perform tests on the material we needed to collect it from the stockpiles. Samples had to be representative of each material deriving from the different processes. The material collected during the period of the research can be quantified in the range of 2000 kg. From now on granite by-products deriving from TBM will be called “AG-1” granite by-products from D&B “AG-2” and granite from Crusher “AG-3”. To sample the granite the UNI EN 932-1 sampling procedure for stockpiles was used. It consists on taking samples from three distinctive areas of the stockpiles (see Figure 22) in which segregation can develop according on the dimension of the aggregate: top, middle and bottom with the aim of having a sample that is representative of the entire stockpile.



**Figure 22. View of the sampling points in one of the stock piles**

Once in the laboratory the collected material was reduced according to the type of test that had to be performed, this was done according to UNI EN 932-2 specification. The quartering method was used and it consists on reducing the sample by successively mixing and dividing into quarters and keeping two opposite quarters of the sample, until the correct amount of material needed to perform specific tests is obtained.



The two procedures just mentioned must be performed correctly as possible in order to have samples representative of the original sample. In Figure 23 is shown material deriving from a quartering method of material deriving from a stockpile of granite obtained from TBM excavation method. These types of procedures were performed for all the three types of material on periodic sessions according on how the excavation progressed to have an overview of the entire material excavated.



**Figure 23. Granite deriving from TBM excavation method**

Once the material was sampled and before performing any type of tests to evaluate the possibility of using such material in road pavement layers it was important to evaluate which type of granite we were dealing with. According to the geological maps of the region, widely explained in chapter 5, and by the information given by geologist the granite had to be a Grandiorite, but to have accurate informations tests on the chemical and mineralogical composition were performed. Such tests were performed on the material deriving from the main and also the service tunnels in order to evaluate the uniformity of the geological body that we were dealing with. To measure the chemical composition of the granite the Inductively Coupled Plasma with Optical Emission Spectrometry (ICP- OES) technique was used, the instrument was a Perkin Elmer Optima 7000 DV ICP-OES Figure 24 available in the laboratories of the Department of Civil Environmental and Architectural Engineering (DICAAR).



**Figure 24. ICP-OES Perkin Elmer Optima 7000 DV (DICAAR)**

The ICP was developed for Optical Emission Spectrometry (OES) in the middle of the 1960s. This system is based on the emission of photons from atoms and ions that have been excited by radiofrequency (RF) induced argon plasma using one of a variety of nebulizers or sample introduction techniques. Liquid and gas samples may be injected directly into the instrument, while solid samples require extraction or acid digestion so that the analytes will be present in a solution. To perform the measurements the granite by-products were reduced to a granular size of 0.063 mm. In Table 1 are reported the average chemical compositions of granite by-product samples collected from stockpiles. Each stockpile derives from the excavation of the main tunnels using D&B excavation method and granite by-products deriving from the service tunnels using TBM method.

**Table 1. Chemical composition of granite by-products deriving from TBM (AG-1) and D&B (AG-2)**

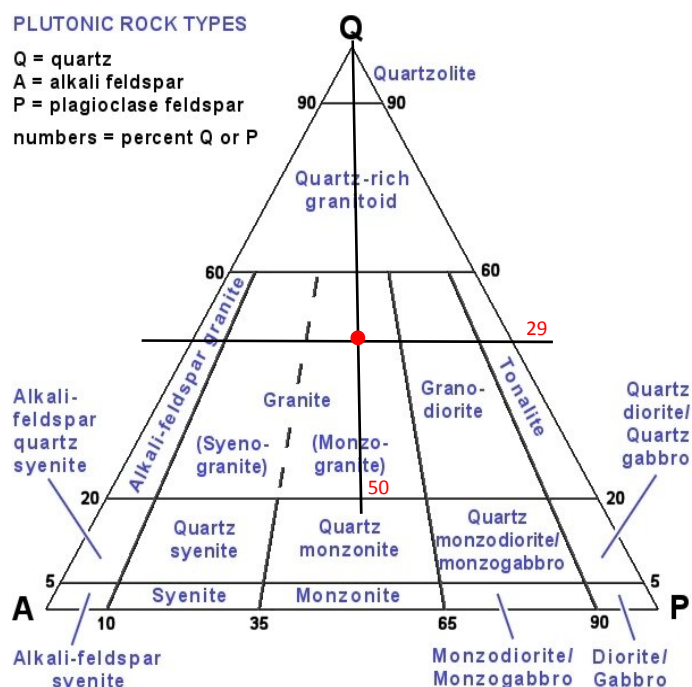
Component	AG-1 (%)	AG-2 (%)
SiO <sub>2</sub>	69.69	66.64
TiO <sub>2</sub>	0.39	0.52
Al <sub>2</sub> O <sub>3</sub>	14.43	14.58
Fe <sub>2</sub> O <sub>3</sub>	3.53	4.46
MnO	0.07	0.08
MgO	1.20	2.07
CaO	2.84	5.09
Na <sub>2</sub> O	3.00	2.82
K <sub>2</sub> O	3.75	3.50
P <sub>2</sub> O <sub>5</sub>	0.12	0.15
LOI	1.55	1.09
Total	100.59	101.04

Plutonic Igneous rocks are classified on their mineral content, the mineral content is expressed as the Mode corresponding to the percentage of each mineral present. The mode is plotted on the QAPF classification diagram also known Streckeisen classification based on the percent of the felsic minerals: Quartz, Alkali Feldspar, Plagioclase and Feldspathoids. With the data reported in Table 1 it is possible to obtain the modal composition of representative AG-1 and AG-2 Table 2.

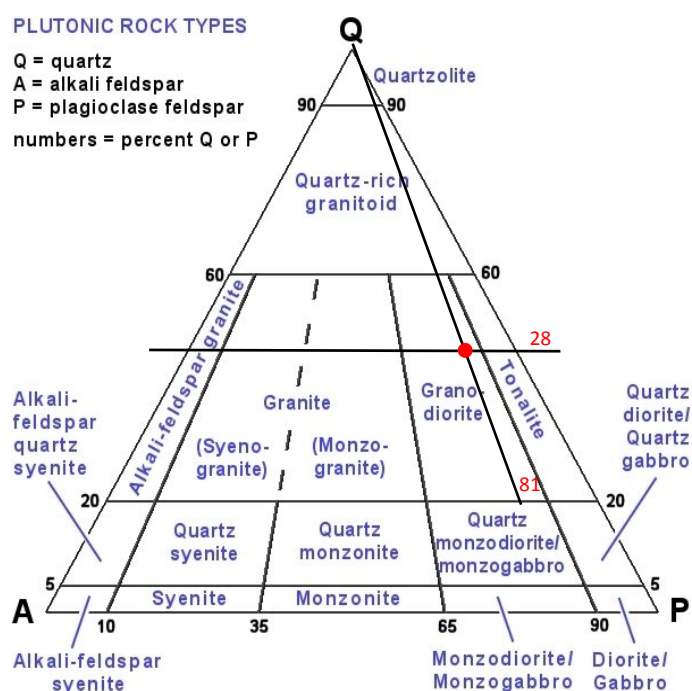
**Table 2. Modal composition of representative AG-1 and AG-2 granites**

Sample AG-1	Vol. %	Sample AG-2	Vol. %
Quartz	25.0	Quartz	22.3%
K-feldspar	30.6	K-feldspar	10.5%
Plagioclase	29.5	Plagioclase	46.6%
Biotite	14.9	Biotite	20.6%

By the modal composition it is possible to plot AG-1 and AG-2 in a QAP classification diagram. Has shown in Figure 25 AG-1 can be referred to a monzogranite while AG-2 can be referred to a granodiorite as shown in Figure 26.



**Figure 25. Modal compositions of the AG-1 plotted in QAP classification diagram**  
(source Andrew Alden, [geology.about.com](http://geology.about.com), reproduced under educational fair use)



**Figure 26. Modal compositions of the AG-2 plotted in QAP classification diagram**  
*(source Andrew Alden, geology.about.com, reproduced under educational fair use)*

To evaluate the mineral composition of the granite by-products, tests were performed using X-Ray Powder Diffraction (XRPD) qualitative analyses. This is a non-destructive technique for the analysis of the crystalline materials, in form of powder or solid. Friedrich and Knipping performed the first x-ray diffraction experiment in 1912 using a crystal of copper sulfate; they obtained a diffraction pattern and concluded that x-ray must be electromagnetic radiation. Each material produces a unique X-ray "fingerprint" of X-ray intensity versus scattering angle that is characteristic of its crystalline atomic structure. It is possible to perform analysis by comparing the XRPD pattern of an unknown material to a library of known patterns performing a search and match procedure conducted on the sample. When X-rays interact with a crystalline substance, one gets a diffraction pattern. The pattern can be used for the identification and quantitative determination of the various crystalline compounds, known as "phases", present in solid materials and powders.

For our study a Rigaku Geigerflex diffractometer was used, operating with CuKalpha radiation, at 30 kV and 30 mA. The comparison of the diffractograms obtained from the samples deriving from the granite by-products were compared with the patterns included in

software database named MDI Jade 5.0. The results obtained on such tests are reported in Figure 27 and Figure 28.

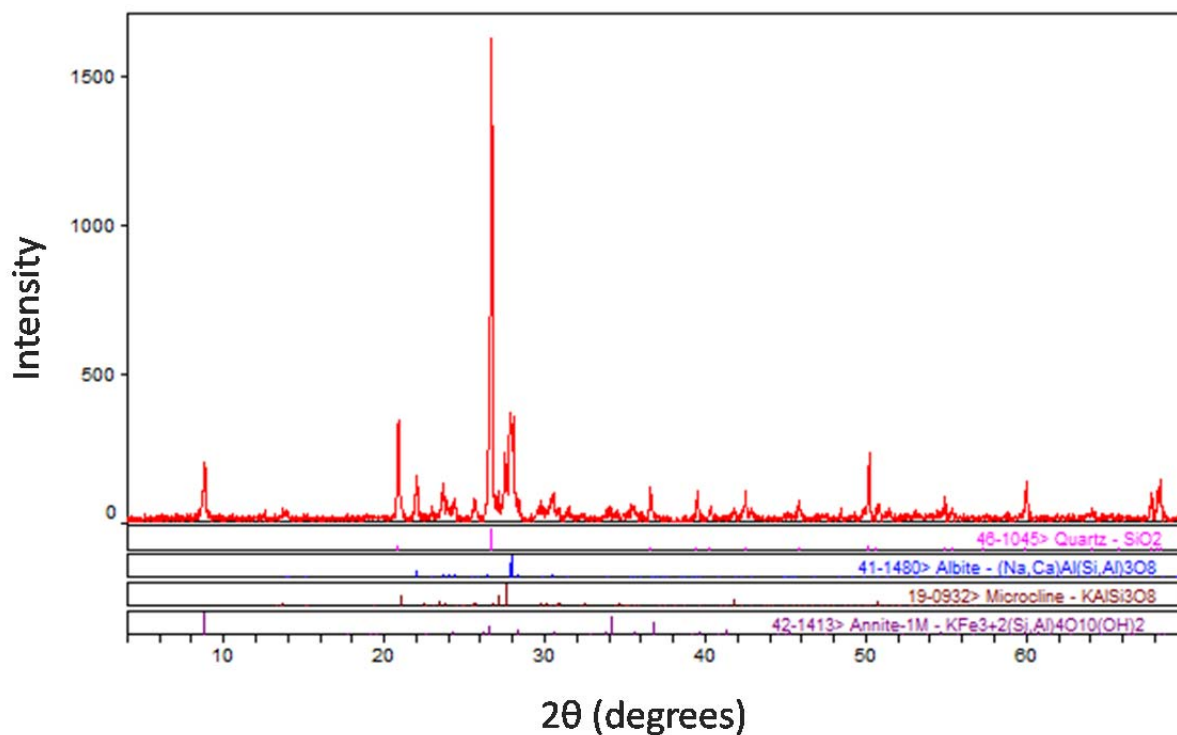
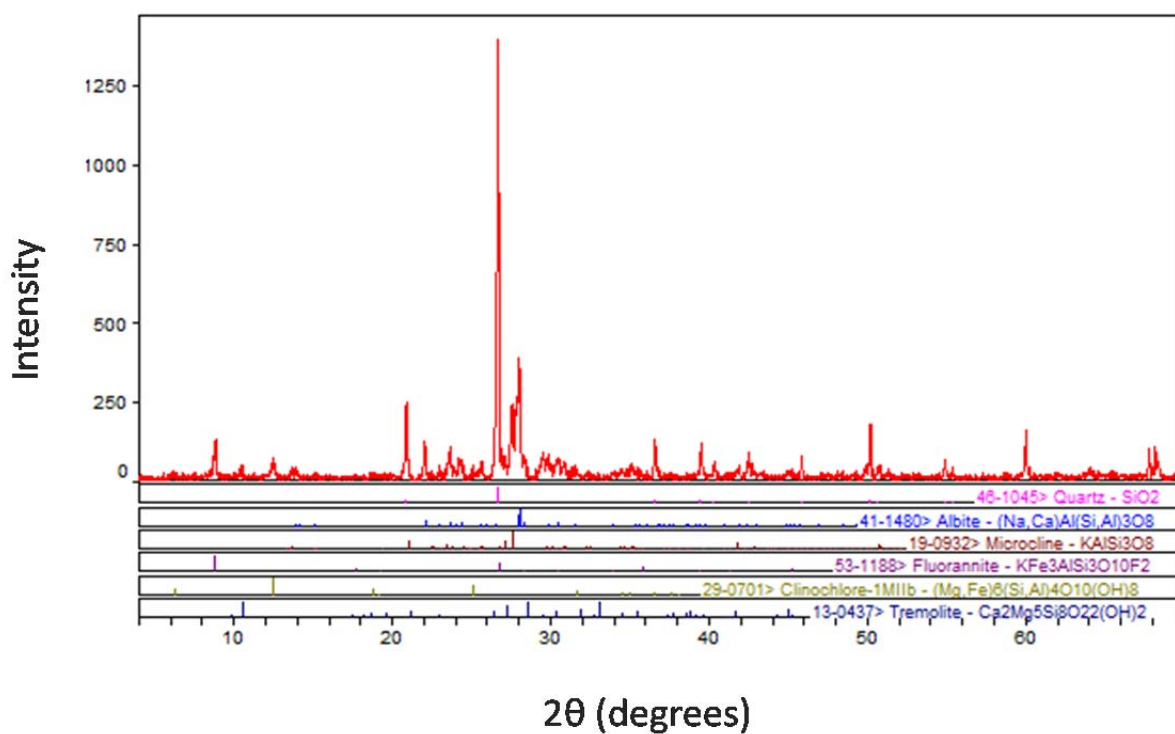


Figure 27. Diffractogram obtained on sample deriving from granite AG-1





**Figure 28. Diffractogram obtained on sample deriving from granite AG-2**

The final test performed in this phase consisted on determine the specific gravity of the aggregates Figure 29.



**Figure 29. Specific gravity weighing apparatus**

The UNI EN 1097-6 specification was used performing hydrostatic weighing on aggregates with particle size between 63 mm and 31.5 mm, while the vacuum Pycnometer (Figure 30) was used to measure aggregates with size ranging from 31.5 mm to 0.063 mm. The tests conducted gave values between 2.65-2.66 g/cm<sup>3</sup>.



**Figure 30. Vacuum Pycnometer**

## **6.2 Environmental Compatibility**

### **6.2.1 Leaching tests**

To evaluate the possibility of using granite by-products for road construction purposes was important to evaluate if such material were environmental compatible. In order to do so tests to evaluate the permissible concentrations of elemental species in the aggregate or their leachates had to be carried out.

Leaching can be defined according to Sharp as “the extraction of a soluble material from an insoluble solid by dissolution in a suitable solvent” (Sharp, 1990). The leaching process involves physical, chemical and biological reactions that mobilize a contaminant but also contributing to the transport mechanism that carry the contaminant away from the matrix. The solvent that induces the leaching process is called the leachant, and the resulting fluid containing the soluble material is named the leachate.

In order to evaluate if the granite by-products deriving from the construction of the tunnels were environmental suitable, leaching tests were performed on several samples deriving from the different processes to which granite was subjected. The Italian legislation and

regulation on this type of matters, that in this last years and months have been changing quite rapidly, have to deal with the LG.D. n.152/2006 and the D.M. n. 161/2012. The LG.D. n.152/2006 the so called “Environmental Code” this Legislative Decree gives the guide lines on which procedure to use to perform the tests and the list of elements (n°22) with the maximum permissible concentration (see Table 3) with also the values of pH that must be in the range of 5.5 and 12.

**Table 3. Maximum permissible concentration of elements**

Elements	Maximum concentrations (mg/l)	Elements	Maximum concentrations (mg/l)
Ni	0.01	NO <sub>3</sub>	50
V	0.25	F	1.5
As	0.05	SO <sub>4</sub>	250
Cd	0.005	Cl	100
Cr	0.05	Cn	0.05
Pb	0.05	Ba	1
Se	0.01	Cu	0.05
Hg	0.001	Zn	3
Asbestos	0.03	Be	0.01
COD	0.03	Co	0.25

The leaching tests must be performed according to the UNI EN 12457-2. The first set of tests was performed following the guidelines of the LG.D. n. 152/2006, but in 2012 the D.M. n. 161/2012 was introduced. This Ministerial Decree regulates terms and conditions of excavated materials giving a new list of basic elements that must be analyzed (see Table 4). In this new Ministerial Decree the elements that must be analyzed are n°12 and no longer n°20 like in the previous Legislative Decree and the maximum permissible concentrations are the same as reported in Table 3. In this new Decree there is the possibility of performing tests on elements not present in the list if there is a doubt that the site from which the aggregates are extracted has been subjected to possible industrial activities or other activities that could have contaminated the site.

**Table 4. List of Elements according to D.M. n. 161/2012**

Elements
As
Cd
Co
Ni
Pb
Cu
Zn
Hg
Hydrocarbons C>12
Total Cr
Cr VI
Asbestos

The maximum permissible concentrations for the elements in Table 4 are those imposed by the LG.D. n. 152/2006. The leaching tests were performed on samples according to UNI EN 12457-2 specifications one stage batch test at a liquid to solid ratio of 10 l/kg with particle size below 4 mm. In our case the sample needed size reduction; this was done using a jaw crusher. In order to eliminate dust and potential contaminants deriving from previous use of the crusher by other types of rocks, several trial sessions using granite were conducted before performing the actual crushing on the material that will be subjected to the leaching test. During the crushing processes it is important to limit excessive crushing in order to prevent the pulverization of the granite. Once the material is reduced to a size below 4 mm it must be subjected to a mixing or agitation procedure. This can be performed by using an end to end tumbler (5 rpm -10 rpm) or a roller bottle table (10 rpm), in this study an end to end tumbler was used as shown in Figure 31. This type of agitation devices was capable of holding 6 vessels of the required size (1000 ml), but to perform the tests 4 vessels were agitated each time. The four vessels were one containing a sample of granite by-product plus a blank sample containing only distilled water to perform a Blank test for the verification of the leaching procedure. The samples were agitated for 24 h at a rate of 6 rpm at a constant temperature of 20°C.



**Figure 31. End to End Tumbler**

Once the sample was agitated for 24 h it had to settle for 15 minutes to let the suspended solids to settle and then it was possible to perform filtration.

#### **6.2.2 Leachate analysis**

Filtration was performed allowing the leachate through 0.45  $\mu\text{m}$  nylon filter paper, using a vacuum filtration apparatus Figure 32. Soon after filtration pH and conductivity was measured and the full chemical analysis was performed.



**Figure 32. Vacuum filtration apparatus**

Two campaigns according to several sets of tests were performed one according to the LG.D. n. 152/2006 analyzing the components reported in Table 3 and the second campaign according to D.M. n. 161/2012 analyzing the components reported in

Table 4. First of all for each test pH and conductivity was measured using a pH meter Figure 33.



**Figure 33. pH meter ready to perform measurements of pH and conductivity**

Results obtained by the measurements are reported in Table 5, it must be reminded that according to the LG.D. n.152/2006 that the pH values must be between 5.5 and 12.

**Table 5. pH and conductivity mean values deriving from tests of each campaign**

Sample	First Campaign		Second Campaign	
	pH	Cond. ( $\mu\text{S}/\text{cm}$ )	pH	Cond. ( $\mu\text{S}/\text{cm}$ )
AG-1	10.01	112.14	9.8	126.3
AG-2	12.08	982	11.62	609
AG-3	9.87	114.00	9.43	162.0
Blank	8.75	37.70	9.16	21.6

The values reported in Table 5 have been used to obtain a chart reported in Figure 34 to better understand the results of each granite by-product and also to compare the two test

campaigns. It is possible to see that pH values are reached in the range of 12 by granite deriving from D&B.

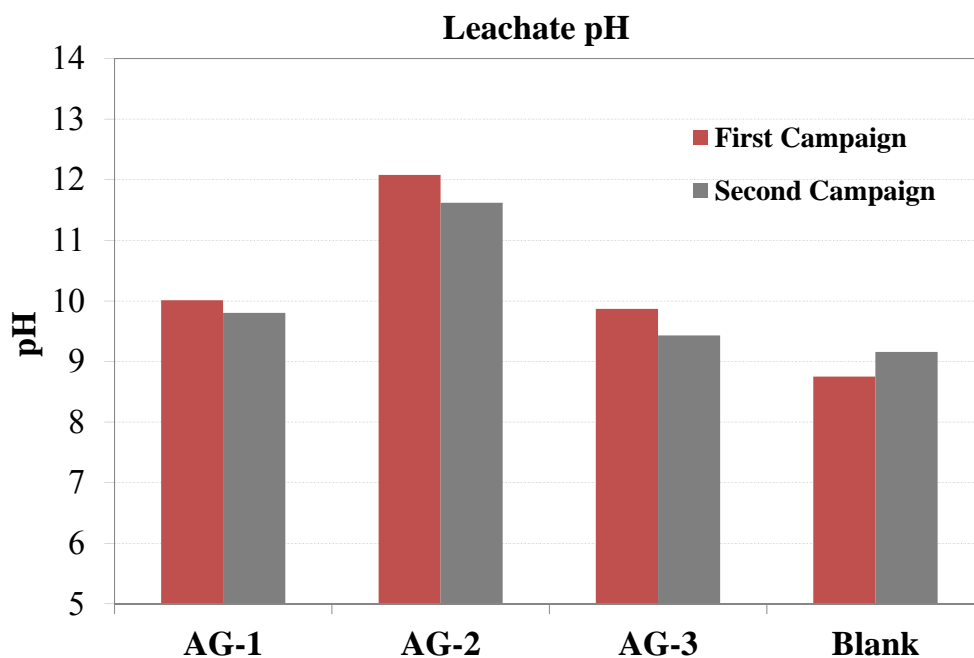


Figure 34. Comparison of pH data values obtained from tests performed in the two campaigns

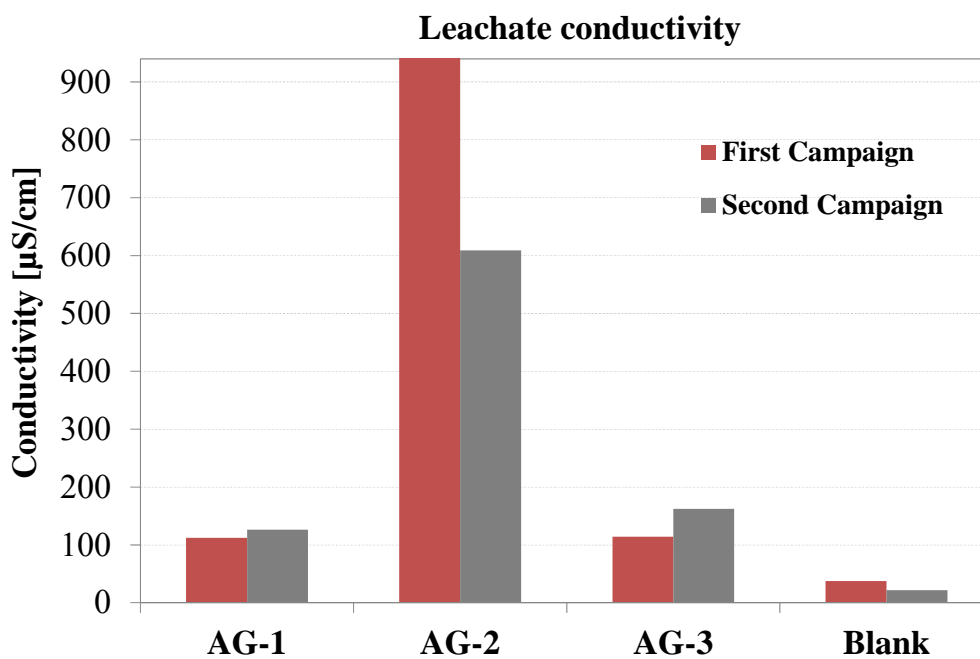


Figure 35. Comparison of conductivity data values from tests performed in the two campaigns

Once pH and conductivity was measured tests were performed on the leachates in order to determine the elements concentration results are reported in Table 6.

**Table 6. First campaign set of test results on elements imposed by the LG.D. n. 152/2006**

Sample	Concentration of leachate (mg/l)			
	$\leq 0.001$	0.001-0.01	0.01-1	1-20
AG-1	Ni Cu Cr Se As Be Co Cd	Pb Zn Ba V	F NO <sub>3</sub>	Cl SO <sub>4</sub>
AG-2	Se As Be Co Cd	Ni Cu Pb	Cr Zn Ba V	F Cl SO <sub>4</sub> NO <sub>3</sub>
AG-3	Cr Se As Be Co Cd	Ni Cu Pb	Zn Ba V	F Cl NO <sub>3</sub> SO <sub>4</sub>
Blank	Ni Cr Se As Be Co Cd Pb V NO <sub>3</sub>	Cu Ba	Zn F Cl SO <sub>4</sub>	

The maximum concentrations of the elements that were analyzed are referred to n°16 of the n°20 elements imposed by the LG.D. n. 152/2006, the four elements that were not analyzed are Asbestos, Mercury (Hg), Cyanide (Cn) and Chemical Oxygen Demand (COD) this due to the lack of instrumentation to measure such parameters. Of the 16 elements analyzed in the first and second campaign only Fluoride (F) was over the maximum concentration. This result was obtained for both AG-2 and AG-3 aggregates.

The tests performed in the second campaign were performed on n°8 of the n°12 elements imposed by the D.M. n. 161/2012. Tests were not performed on Asbestos, Mercury (Hg), Chrome (Cr IV) and Hydrocarbons with C>12, this due to the lack of instrumentation. The results of such tests are reported in Table 7.



**Table 7. Second set of tests on elements imposed by D.M. n. 161/2012**

Sample	Concentration of leachate (mg/l)			
	$\leq 0.001$	0.001-0.01	0.01-1	1-20
AG-1	Cr Co Cd	Ni Pb	As Cu Zn F NO <sub>3</sub>	Cl SO <sub>4</sub>
AG-2	Co	Cd Ni	Cu Cr Pb Zn	As F Cl SO <sub>4</sub> NO <sub>3</sub>
AG-3	Ni Cr As Co Cd Pb	Cu	Zn	F Cl NO <sub>3</sub> SO <sub>4</sub>
Distilled Water	Ni Cr As Co Cd Pb NO <sub>3</sub>	Cu	Zn F Cl SO <sub>4</sub>	

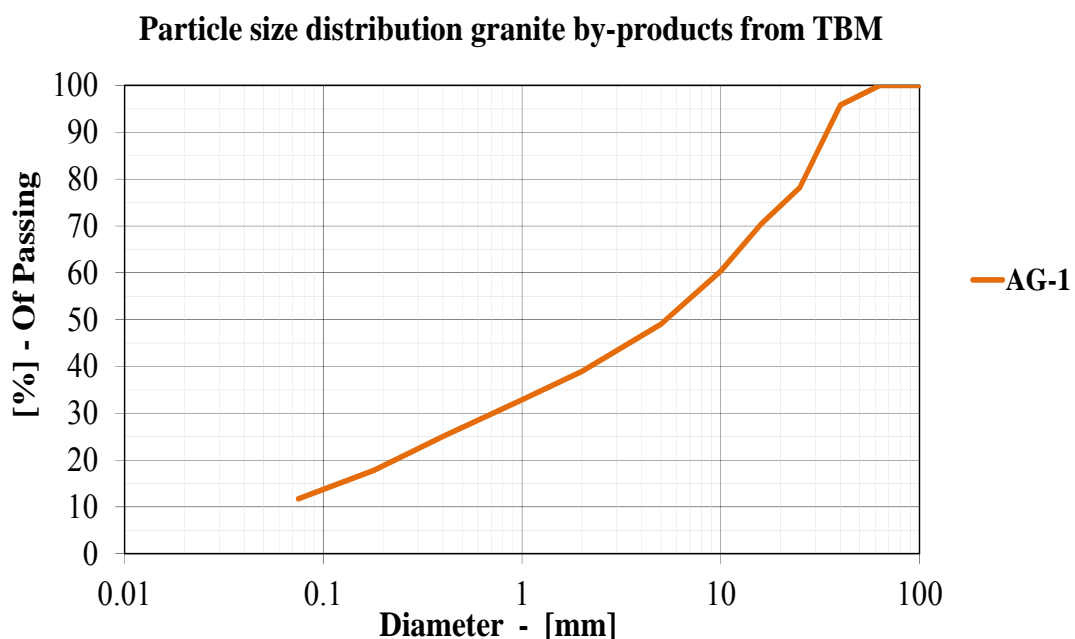
### 6.3 Test on granite by-products to be used in unbound granular layers

Once tests to assess the environmental compatibility were conducted and results obtained a next phase of the research started. Granite by-products aggregates were tested to evaluate the possibility of using this type of material in road pavement layers. In this research the evaluation of such materials started from the bottom layers, analyzing the use of granite by-products to be used as aggregates in unbound layers. To evaluate the possibility of using granite by-products in unbound granular layers it was important to evaluate the performance according to the imposed specifications. It was chosen to follow the specifications imposed by the “Ministry of Infrastructure and Transport of Italy”. In the following lines will be discussed the tests conducted.

Test were performed to evaluate particle size distribution of the granite by products deriving from: TBM (AG-1), D&B (AG-2), Crusher (AG-3), in order to evaluate if the gradation was inside the envelope imposed by the specifications, and to verify in which way different processes modify the gradation of the aggregates. **The gradation** of aggregates is one of the factors that most agencies use on the selection of the aggregates. Also **Petrographic examination** to evaluate the mineralogical makeup and geological source of aggregates, have been shown to have significant impact on performance. Petrographic examination is recommended as a validation test because of its ability to distinguish between aggregate types, mineralogical make-up, and general indications as to performance potential.

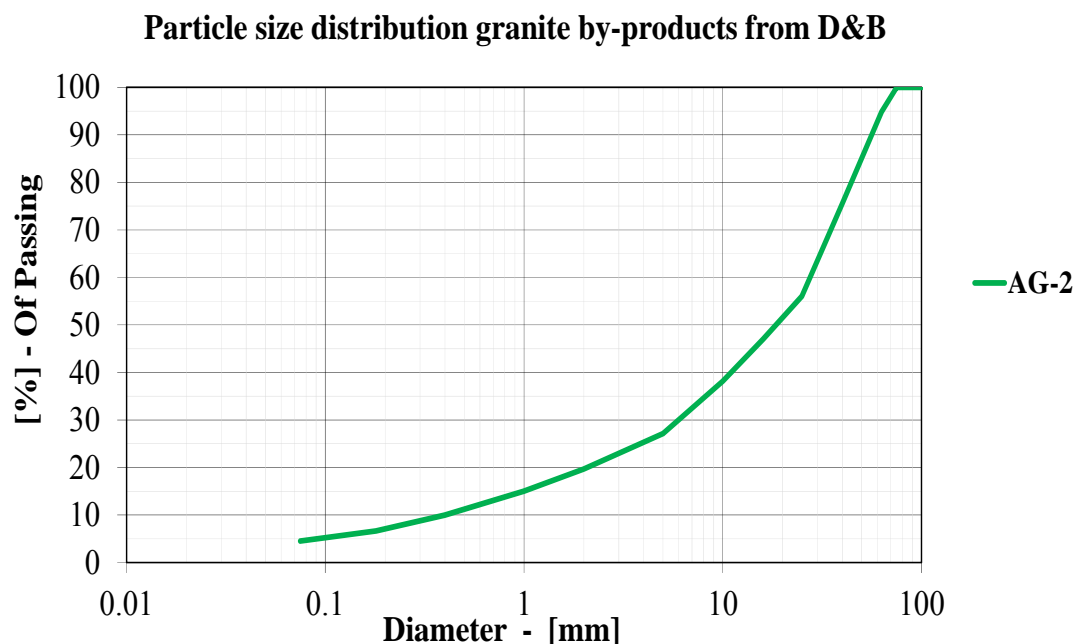
**Atterberg limits** are also important validation tests to classify aggregates. **Specific gravity** it is not and indicator of quality itself but it can be an indicator of potential problems. **Moisture and density relationship** obtained by performing laboratory compaction tests to determine important information's like density, useful to anticipate achievable values in field performance and also to prepare laboratory specimens for other tests. Compaction of aggregate materials generally results in increases in density, shear strength, and stiffness and decreases permeability **Los Angeles abrasion test** has long been used by many agencies as an index for aggregate toughness. **California Bearing Ratio (CBR)** the CBR test (soaked) is recommended as a validation test because of its widespread use as a strength parameter in pavement structural design and because of its long-term historical acceptance as an indicator of performance. CBR tests on granular materials must be conducted with extreme caution, and several samples will be used to ensure satisfactory and accurate results.

The determination of particle size distribution was obtained by performing tests according to UNI-EN 933-1 specifications (UNI-EN 933-1, 2009). Tests were performed on various samples collected during different periods depending on the advance and progress of the excavation of the tunnels. In Figure 36 is reported the average particle size distribution obtained from the samples collected deriving from stockpiles of granite AG-1.



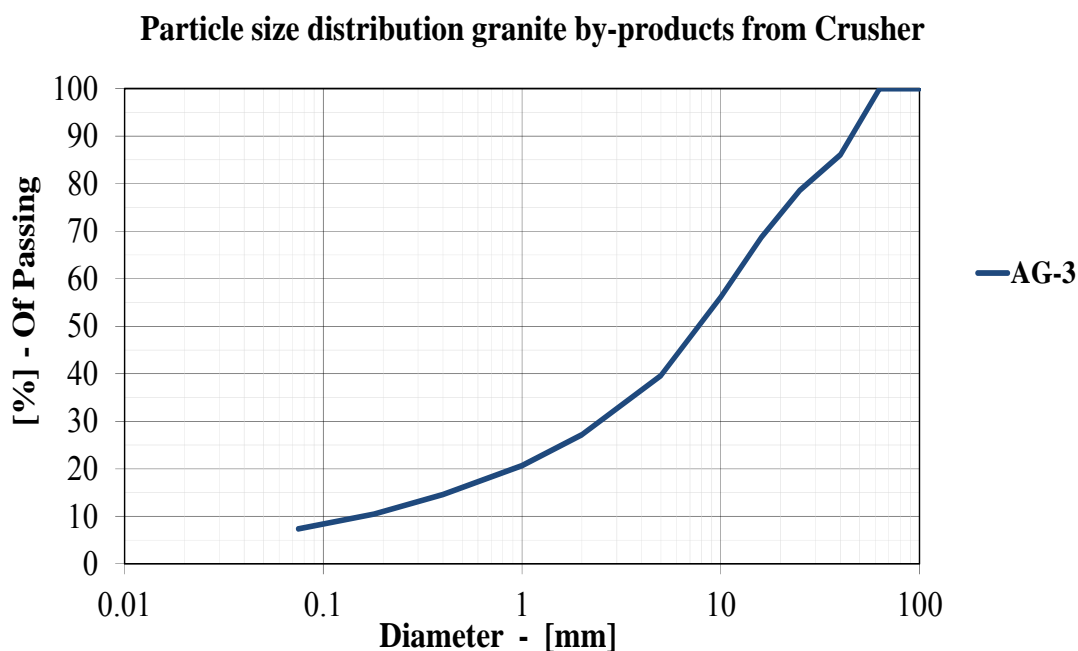
**Figure 36. Average Particle size distribution on tests conducted on granite AG-1**

In Figure 37 is reported the average particle size distribution obtained from samples collected during the campaign of the research on stockpiles of granite AG-2.



**Figure 37. Average Particle size distribution on test conducted on granite AG-2**

In Figure 38 is reported the average particle size distribution obtained from samples deriving from the crushing of the larger blocks deriving from D&B.



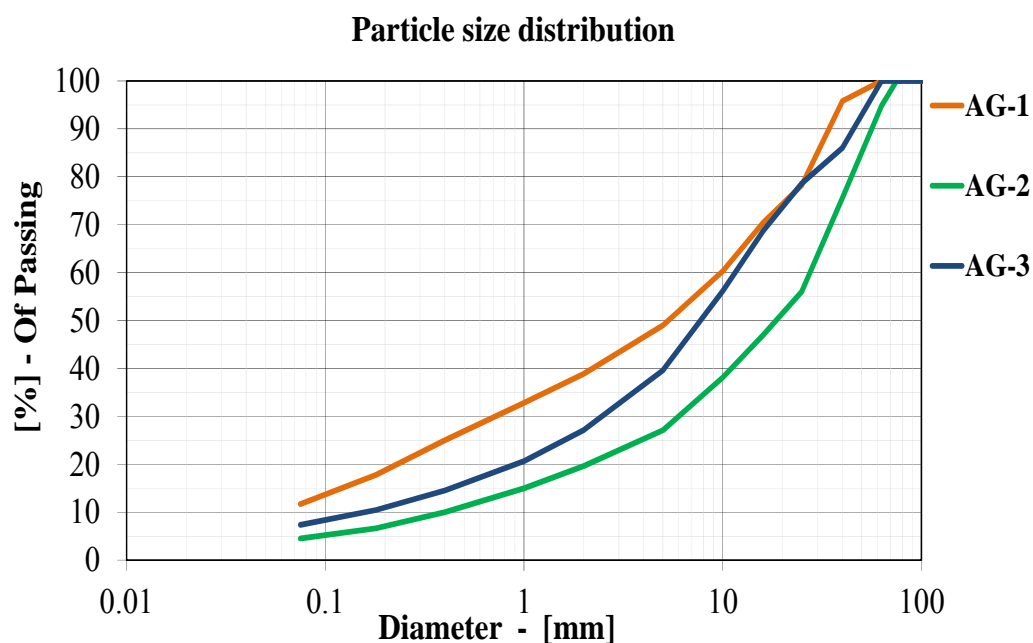
**Figure 38. Average Particle size distribution on test conducted on granite AG-3**

In Table 8 are reported the standard deviation values according to all the samples collected for each of the three types of granite by-products.

**Table 8. Standard Deviation of the three types of granites analysed**

Grain Diameter (mm)	Stand. Deviation AG-1 (%)	Stand. Deviation AG-2 (%)	Stand. Deviation AG-3 (%)
100	0.00	0.00	0.00
75	0.00	0.00	0.00
63	0.00	7.30	0.00
40	2.51	1.62	4.85
25	2.55	7.33	3.22
16	2.23	8.58	3.41
10	2.96	7.02	3.28
5	2.24	4.62	3.56
2	2.25	3.29	3.33
1	2.30	2.37	2.59
0.4	2.20	1.40	1.61
0.18	2.18	0.91	1.04
0.075	2.19	0.68	0.82

In Figure 39 are shown in comparison the three curves of the average particle size distribution of the three granite by-products aggregates.



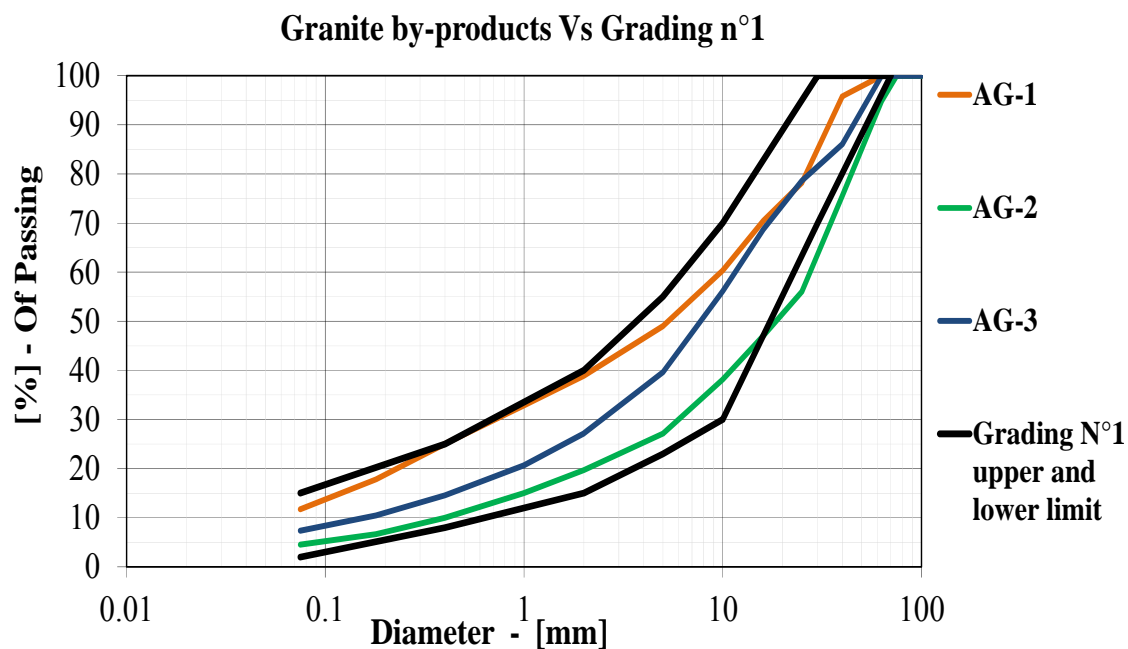
**Figure 39. Comparison between the three granite by-products**

The three average particle size distribution curves must be compared with values imposed by the Italian “Ministry of Infrastructure and Transport”. That imposes two types of gradings for unbound granular layers that can be used in foundation and base layers. In Table 9 are reported the upper and lower limits for the two gradings imposed by the “Ministry of Infrastructure and Transport”.

**Table 9. Upper and lower limit for the two types of gradings**

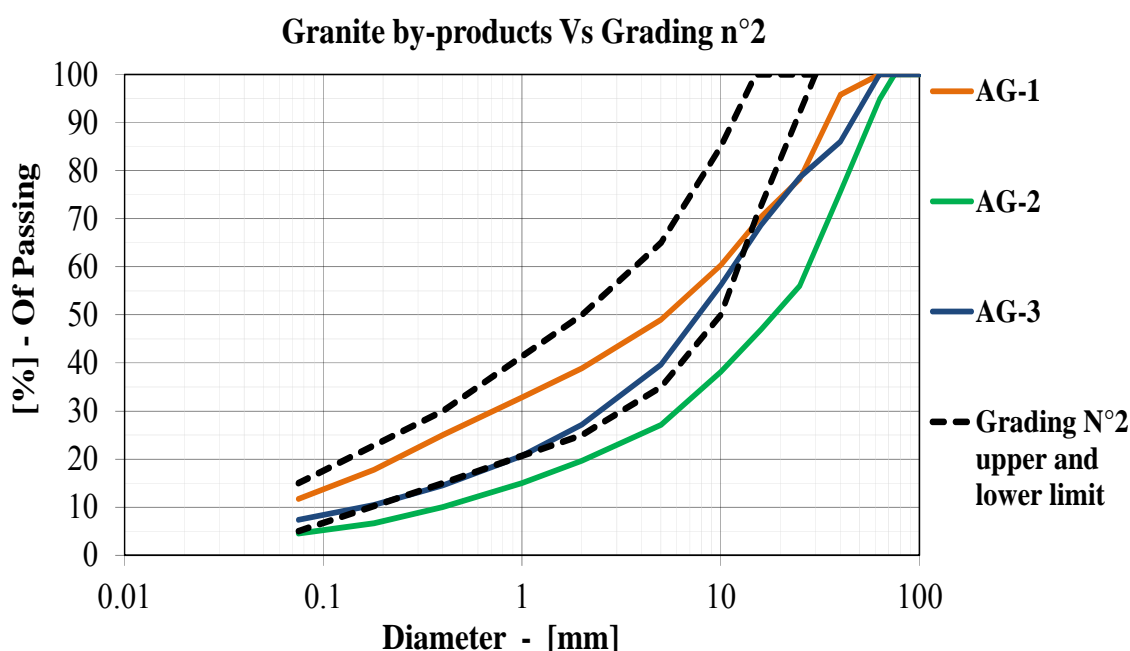
Grain Diameter (mm)	Grading n°1	Grading n°2
70	100	-
30	70 - 100	100
15	-	70 - 100
10	30 - 70	50 - 85
5	23 - 55	35 - 65
2	15 - 40	25 - 50
0.4	8 - 25	15 - 30
0.075	2 - 15	5 - 15

In Figure 40 is shown the comparison between the upper and lower limit of grading n°1 that can be used in foundation and base layers, and the three types of granite by-products.



**Figure 40. Granite by-products Vs grading n°1**

In Figure 41 is shown the comparison between the upper and lower limit of grading n°2, and the three types of granite by-products.



**Figure 41. Granite by-products Vs grading n°2**

It is possible to see that in Figure 40 the three grading of the granite by-products are almost inside the grading envelope, only granite from D&B is slightly out of the envelope imposed. In Figure 41 all the three grading are outside the grading envelope. Other tests considering petrographic examination, previously reported, were conducted and gave us results on the chemical and mineralogical composition (see Table 1 and Table 2) and determining aggregate type: AG-1 deriving from the granite family of monzogranites while AG-2 and also AG-3 from the granite family of granodiorites. The specific gravity for all three types of granite by-products was of  $2.66 \text{ g/cm}^3$ .

Atterberg limit tests were performed according to CNR-UNI 10014 (CNR-UNI 10014, 1969). Performing this test it is possible to determine the liquid limit and the plastic limit.

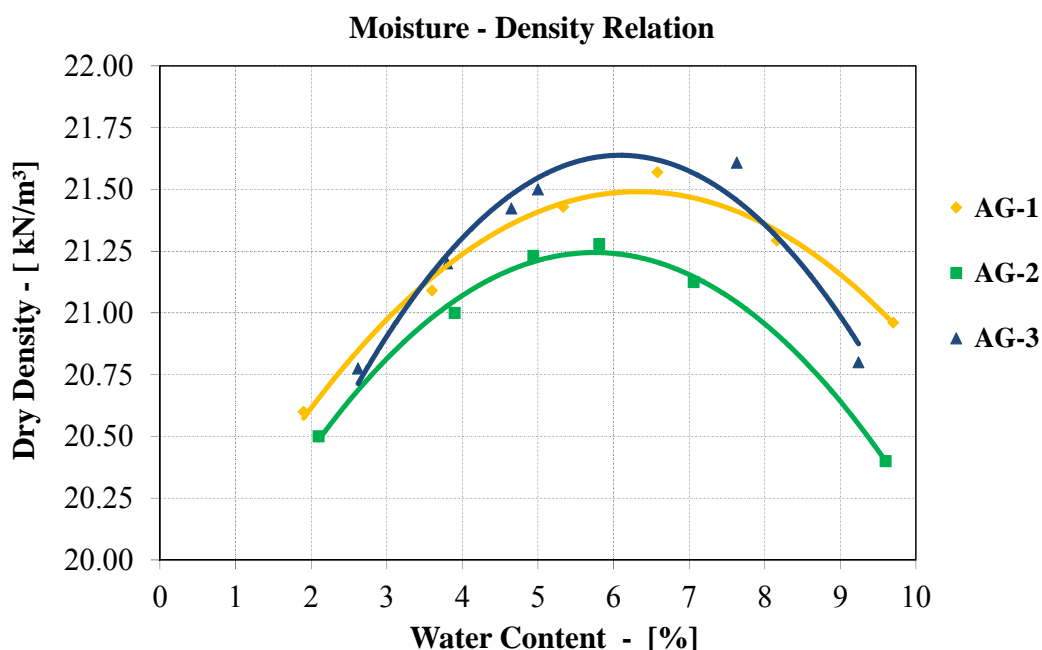


**Figure 42. Determination of the plastic limit**

The three types of granite by-products resulted non plastic. Moisture and density relationship was measured performing modified Proctor test using UNI-EN 13286-2 specifications (UNI-EN 13286-2, 2010).

This European standard specifies tests methods for the determination of the relationship between the water content and the dry density of hydraulically bound or unbound mixtures after compaction under specified test conditions using Proctor compaction. Material with selected moisture content is placed in cylindrical mould (150 mm) and compacted using a compactive effort of controlled magnitude. The material is compacted into the mold to a certain amount of equal layers (n°5), each receiving a number blows from a standard weighted hammer at a specified height. This process is then repeated for various moisture contents in this case n°6 and the dry densities are determined for each one of them. The graphical relationship of the dry density to moisture content is then plotted to establish the compaction curve. The maximum dry density is finally obtained from the peak point of the compaction curve and its corresponding moisture content, also known as the optimal moisture content. In the tests conducted samples were prepared using each of the three granite by-products and mixing each of the aggregates with six selected water content values: 2%, 4%, 5%, 6%, 8% and 10%. The average values obtained on tests conducted on the three granite by-products are reported in Figure 43 in which moisture content is related to dry density and it is possible to obtain the optimum water content.





**Figure 43. Moisture - Density relationship for the three tested granite by-products**

From the chart reported in Figure 43 it is possible to obtain the peak points of the compaction curve for each of the three granite by-products determining optimum water content and the related dry density Table 10.

**Table 10. Optimum water content and dry density values for the three types of granite by-products**

Sample	Dry density (kN/m <sup>3</sup> )	Optimum water content (%)
AG-1	21.55	6.5
AG-2	21.30	5.4
AG-3	21.65	6.0

It is reasonable to state that the values obtained are possibly related to the particle size distribution of the three aggregates, the presence of more fines in granite from TBM (AG-1) and from crusher (AG-3) can be considered the reason of higher values of moisture content and dry density. Fine aggregates as well as absorbing more water tend to fill the voids in the coarse aggregate and act as a workability agent improving dry density values.

Los Angeles abrasion test was performed using UNI EN 1097-2 specifications (UNI-EN 1097-2, 2010). Los Angeles abrasion test measures the deterioration of standard gradation of mineral aggregates through abrasion and impact. It consists on preparing a certain amount of aggregates with a determined size fraction that is located in a rotatable drum in which still balls are placed and must perform a number of rotations. After being subjected to the rotating drum, the weight of aggregate that is retained on the 1.60 mm sieve is subtracted from the original weight to obtain a percentage of the total aggregate weight that has broken down and passed through the 1.60 mm sieve.

The Los Angeles standard test parameters: specimen of  $(5000 \pm 5)$  g with size fraction 10/14 mm aggregates, eleven steel balls each of 45–49 mm in diameter and weighing in total 4690–4860 g, rotation of the steel drum 500 revolutions at a constant speed rotational of between  $31\text{--}33\text{ min}^{-1}$ .

The standard test method allows also using other aggregate fractions alternative to the standard 10/14 mm size, in fact in this research the size fraction used was of 4/8 mm, this involves a variation of the number of balls passing from eleven to eight and on the total weight that must be between 3410–3540 g. The material after fragmentation see Figure 44 is washed and sieved using a 1.6 mm sieve, the material retained is oven dried and weighed.



**Figure 44. Material after Los Angeles abrasion test sample AG-1**

The Los Angeles Coefficient LA is calculated by the formula:

$$LA = \left( \frac{5000 - m}{50} \right) \text{ UNI-EN 1097-2: 2010}$$

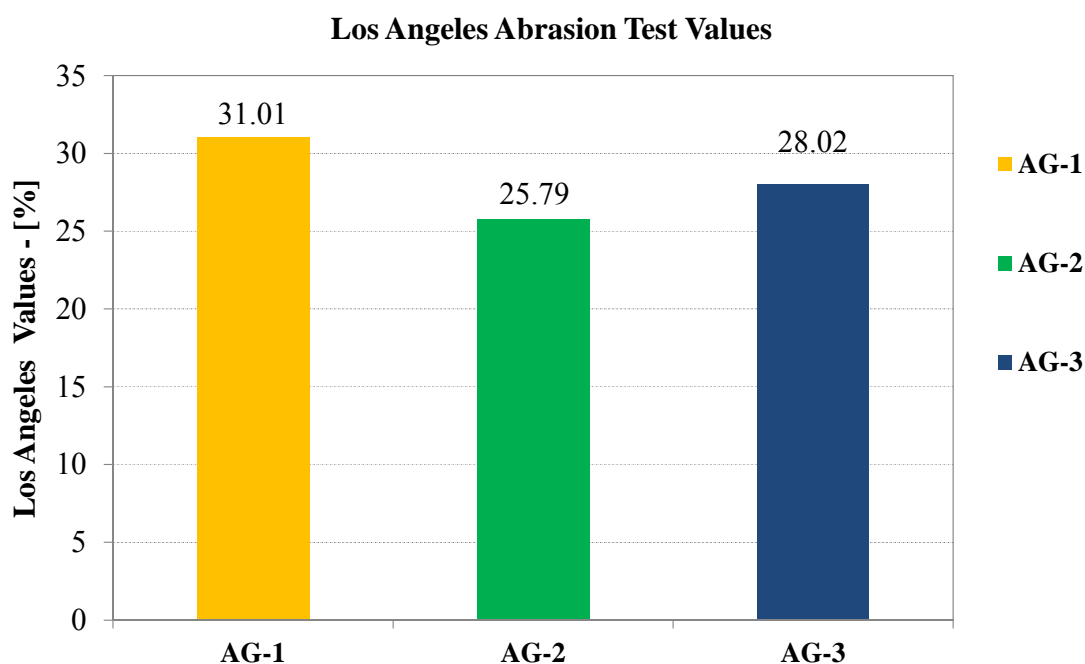
$m$ : is the amount of aggregate in grams retained on the 1.6 mm sieve after fragmentation

As reported in Table 11 the average Los Angeles abrasion values that have been measured for the granite by-products are respectively: 31.28% for aggregates AG-1, 26.18% for those AG-2 and 28.76% for AG-3. The higher value obtained for AG-1 samples can be explained due to the shape that is flat and elongated facilitating the aggregates to disaggregate more easily.

**Table 11. Los Angeles abrasion test values**

Sample	Los Angeles Abrasion Value (%)	Standard deviation (%)
AG-1	31.01	0.86
AG-2	25.79	1.46
AG-3	28.02	1.07

In Figure 45 are reported in a chart the values obtained from the tests conducted performing Los Angeles abrasion test.



**Figure 45. Los Angeles abrasion test values on the three types of granite by-products**

In order to be able to measure the California Bearing Ratio (CBR) that is a test method for the determination of immediate bearing style and linear swelling the following specification was used UNI-EN 13286-47 (UNI-EN 13286-47, 2006).

CBR is the ratio of force per unit area required to penetrate a soil mass with standard circular piston at the rate of 1.27 mm/min, to that required for the corresponding penetration of a standard material. The CBR test was developed initially for the evaluation of the laboratory and in-situ subgrade strength. Presently, the laboratory CBR test is used throughout the world as a quick means of characterizing qualitatively the bearing capacity of soils and unbound base and sub-base materials. This type of test was developed by the California Department of Transportation before World War II.

Parameters and procedures to perform the test are the following:

- After sieving the material on a 22.4 mm sieve, prepare approximately 7.5 kg of mixture with the chosen water content, using percentages already established by the results obtained by the modified proctor test.
- The dimension of the moulds will be that of the modified proctor tests. A spacer disc is placed over the base plate. In Figure 46 are shown the mould the extension collar that is placed on top of the mould and the space disc; the mixture will be placed inside the mould in three different layers each layer will be compacted with 56 blows each.
- After compaction remove the spacer disc from the mould, place a filter paper on the perforated baseplate. Invert the mould containing the compacted specimen, and clamp the baseplate to the mould so that the specimen is in contact with the filter paper. At this step it is possible to place filter paper on top of the specimen followed by the perforated top plate and the fitting of the requisite number of annular surcharge discs around the stem on the perforated plate.
- At this step it is possible to place the specimen in an immersion tank filled with water, to a level that allows free access of water to the top and bottom of the specimen. To measure vertical expansion a measuring device is mounted. Initial reading for swell must be performed and allow the specimen to soak for a minimum of 96 h. Maintaining a constant water level during this period.
- After that the specimen has been soaked for at list 96 h it is possible to perform the tests for the Californian bearing ratio. The specimen is placed on the testing machine (Figure 47) where a load is applied at a constant rate of 1.27 mm/min.

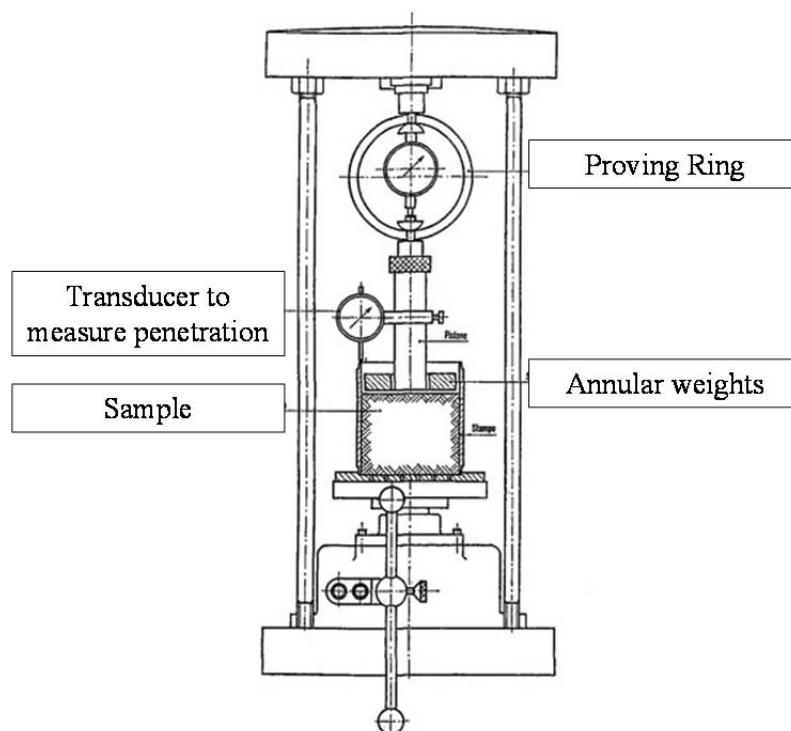


**Figure 46. Mould, extension collar and spacer disc**



**Figure 47. Testing machine to perform CBR tests**

Recording must be done of the load readings (proving Ring) and of penetration of the piston using a transducer. In Figure 48 are reported the main features of the testing apparatus.



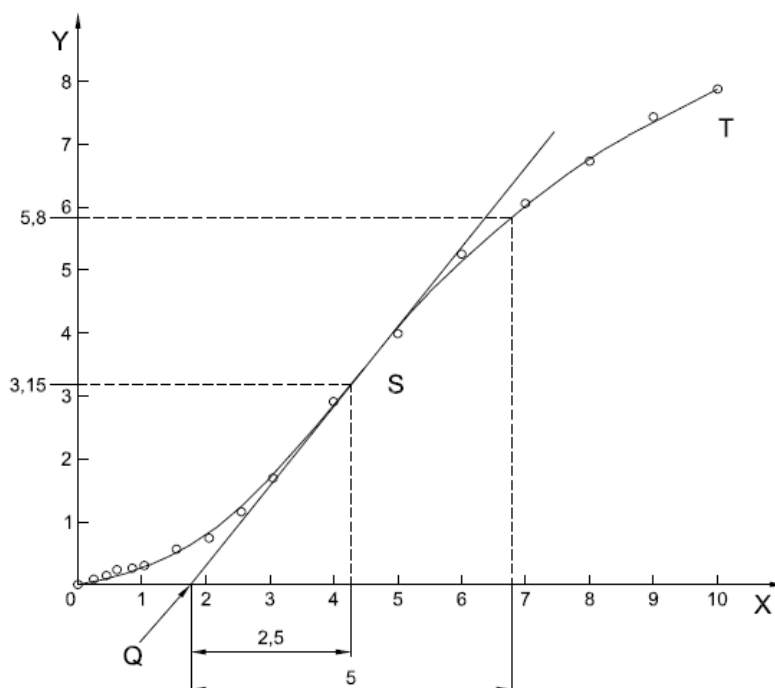
**Figure 48. Californian bearing ratio testing apparatus**

For the calculation and the expression of results the following guidelines according to specifications must be followed.

A force/penetration curve must be plotted; the plot will be formed by plotting each of the values of force as ordinate against the corresponding penetration as abscissa drawing a smooth curve through the points. The normal type of curve is convex upwards and needs no correction.

If the initial part of the curve is concave upwards Figure 49, a correction shall be made by drawing a tangent to the curve at the point of the greatest slope (i.e. the point of inflexion, S).

X = Penetration in mm; Y = Force in kN; Q = New origin.

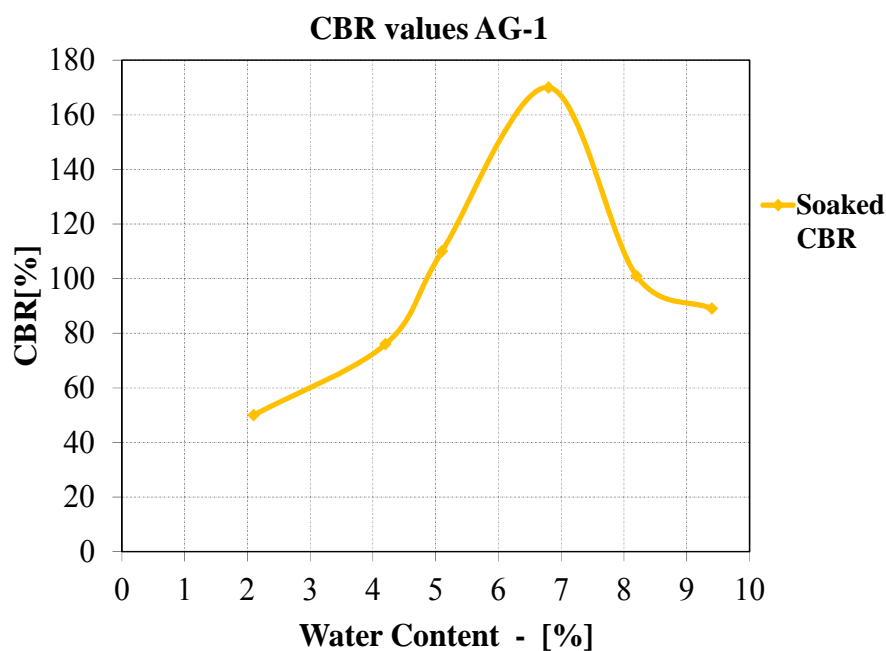


**Figure 49. Force/penetration curve case of the correction of the origin**  
(source EN-UNI 13286-47)

The new origin ( $Q$ ) will be intersection of the tangent with the penetration axis. The correct force/penetration curve to be used in the calculations shall be this tangent from where it cuts the penetration abscissa to its point of contact with the curve and hence the curve itself. The corrected curve shall be represented by  $QST$ , with its origin at  $Q$ , from which a new penetration scale can be marked Figure 49.

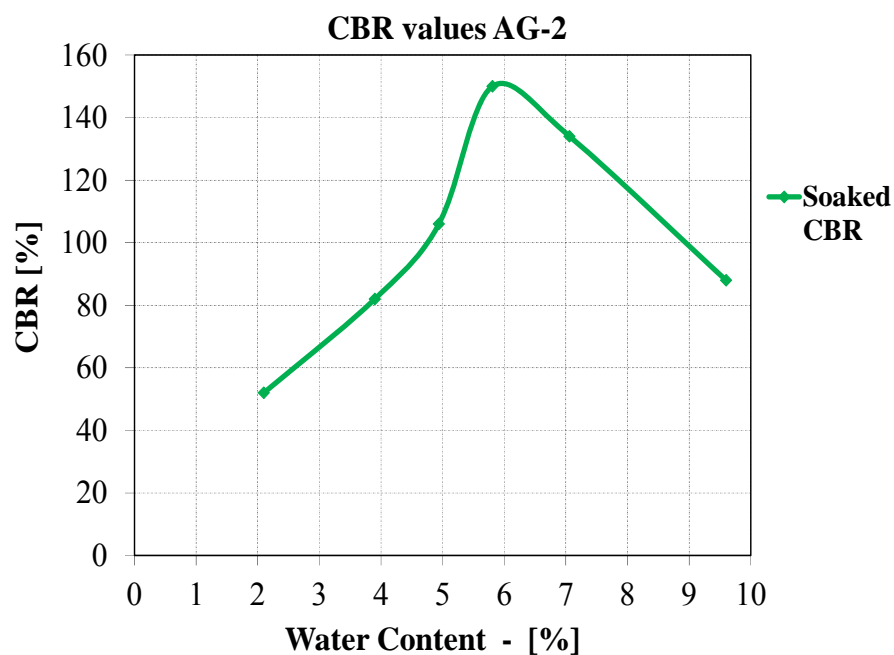
The calculation of the Californian bearing ratio is obtained from the test curve (with corrected penetration scale if appropriate) by reading off the forces in kN corresponding to the 2.5 mm and 5 mm penetration. The values of forces obtained must be expressed as a percentage of the reference forces at these penetrations: 13.2 kN and 20 kN respectively.

CBR tests were conducted on all three types of granite by-products for each of the aggregates no swelling was measured. When plotting the values each of the obtained curve had to be always corrected. In Figure 50 are reported the mean values of CBR, for tests conducted on aggregates AG-1 type.



**Figure 50. Moisture-CBR relation for tests conducted on granite by-products AG-1**

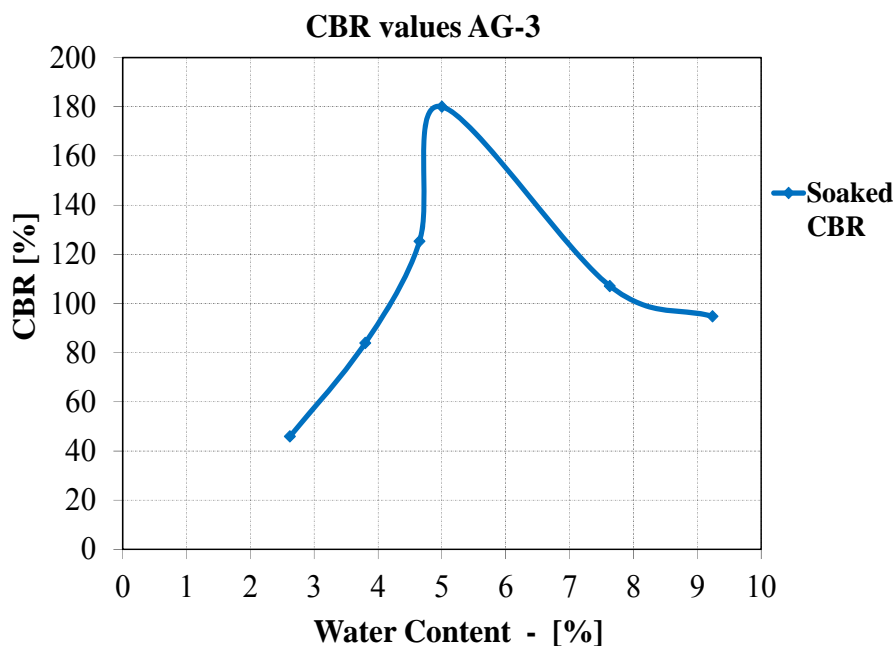
In Figure 51 are reported the mean values on aggregates AG-2 type.



**Figure 51. Moisture-CBR relation for tests conducted on granite by-products AG-2**



In Figure 52 are reported the mean values of the tests conducted on aggregates AG-3 type.



**Figure 52. Moisture-CBR relation for tests conducted on granite by-products AG-3**

Sand equivalent test was performed according to UNI-EN 933-8 specifications (UNI-EN 933-8, 2000). In Italy it is possible to perform this test also according to CNR B.U. 27/72 specifications; the two tests procedures are very similar. This European Standard specifies a method for the determination of the sand equivalent value of the 0-2mm fraction in fine aggregates and all-in aggregates. A test portion of aggregates (0-2mm) and a small quantity of flocculating solution are poured into a graduated cylinder and are left for ten minutes to soak the test specimen. At the end of the ten minutes the cylinder must be sealed and placed on the shaking machine Figure 53.

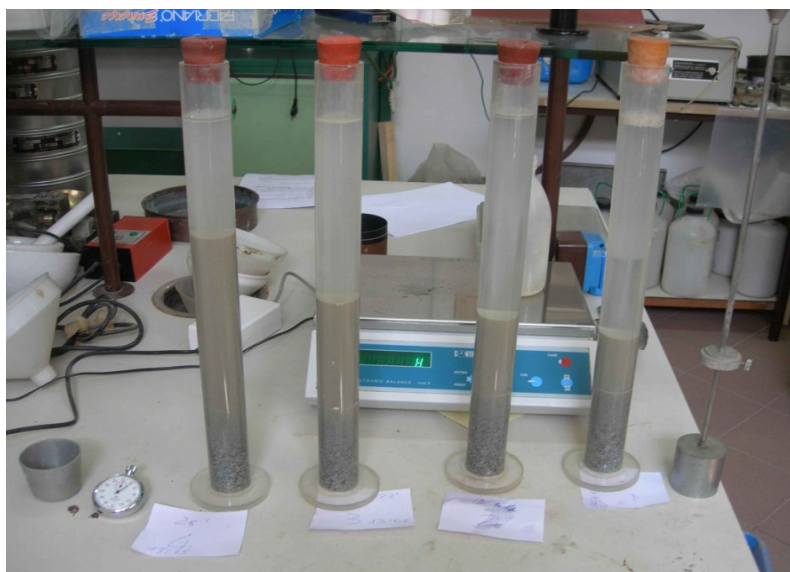
The cylinder must shake for 30 sec. after the shaking the cylinder can be placed in a vertical position. The rubber bung that was used to seal the cylinder is removed and the washing procedure can take place.



**Figure 53. Shaking Machine**

The washing procedure is divided in different steps:

- rinsing the walls of the cylinder using the washing solution
- the washing solution must be used to agitate the contents and encourage the fines and clayey components to rise upwards
- slowly and regularly the washing tube must be raised
- when the level of liquid approaches the upper engraved mark slowly lift the washing tube and regulate the flow so as to maintain the level of liquid at the upper mark until the tube has been withdrawn entirely and the flow stopped
- Once the washing tube has been withdrawn it is possible to start the settling period Figure 54.



**Figure 54. Sand Equivalent test settling period**

For the measurements the graduate cylinder must be left without disturbance and free from vibration for 20 minutes.

At the end of this period, the sand equivalent value (SE) is calculated as the height of sediment expressed as a percentage of the total height of flocculated material in the cylinder. In Table 12 are reported the mean values of the tests conducted to determine sand equivalent value for the three granite by-products.

**Table 12. Sand Equivalent Test Values**

Material	Sand Equivalent Value (%)
AG-1	40.81
AG-2	39.92
AG-3	37.02

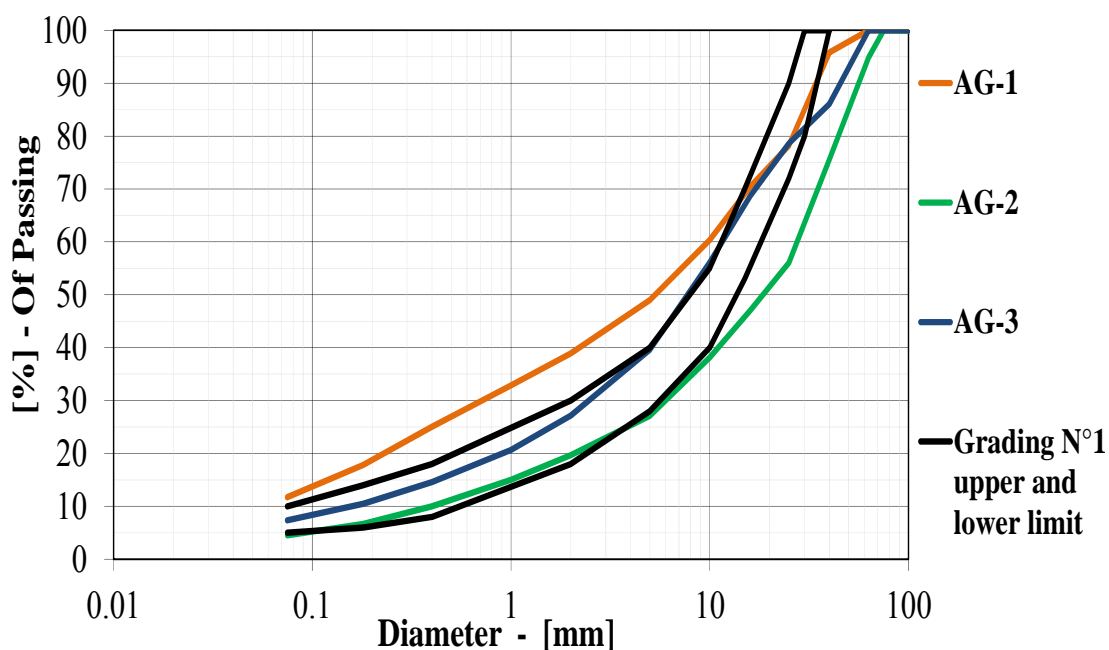
## 6.4 Test on granite by-products to be used in hydraulic bound layers

Tests were performed in order to evaluate the possibility of using granite by-products for the construction of cement bound granular layers. In order to determine compressive strength values on the prepared mixtures; a total of n°40 samples were prepared to evaluate the performance following the specifications imposed by the C.N.R. 29/72 protocol. Tests were performed on two types of granite by-products AG-1 n°20 samples and AG-2 n°20 samples. The specimens were molded using five different percentages of cement contents ranging from 0.5%, 1.5%, 2%, 3% and 3.5% according the specifications it is possible to use cement content up to 5%. Samples were prepared using first granite by-products AG-1. For each chosen percentage of cement, preliminary tests were performed to obtain optimum moisture content and maximum density. The initial water content that was used was that obtained from the modified proctor tests, in the case of AG-1 samples optimum water content resulted in an average value of 6.5%. This value was gradually increased when increasing the percentage of cement in the mixtures. Same procedure was adopted when preparing specimen of soil-cement mixtures using granite by-products AG-2, in this case the initial water content was 5.4%. Before performing any tests the right grading was needed to prepare the specimen. C.N.R. 29/72 specification imposes two gradings one for major Highways (grading n°1) and the other for minor highways (grading n°2). In Table 13 are reported the gradings imposed by C.N.R. 29/72 specification.

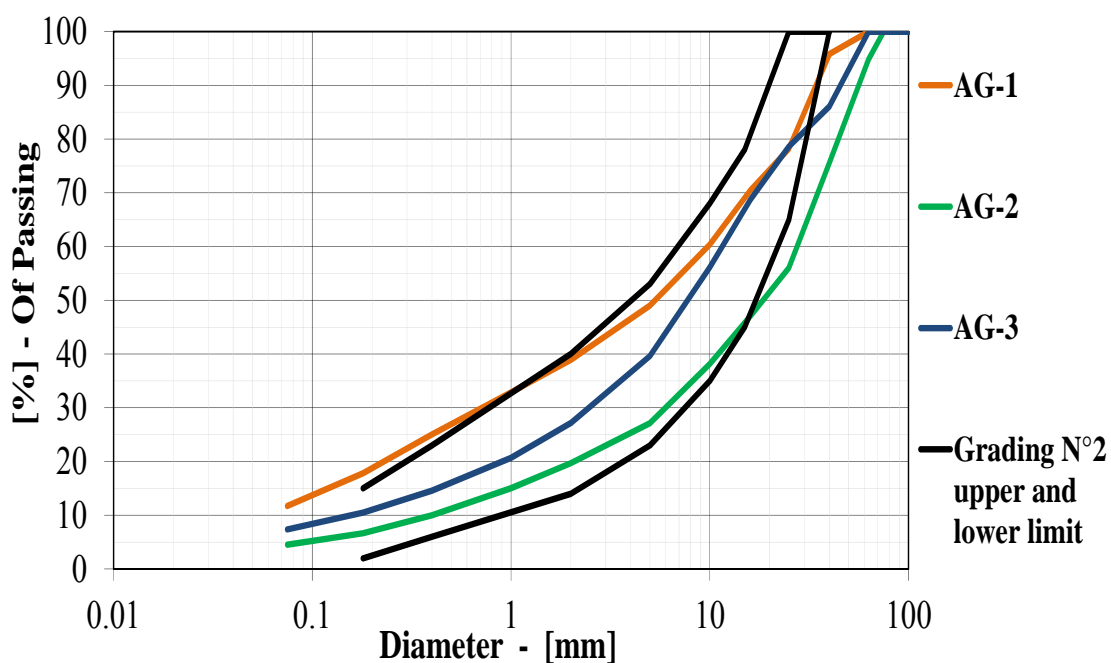
**Table 13. Gradings imposed by C.N.R. 29/72**

Grain Diameter (mm)	Grading n°1	Grain Diameter (mm)	Grading n°2
40	100-100	40	100-100
30	80-100	25	65-100
25	72-90	15	45-78
15	53-70	10	35-68
10	40-55	5	23-53
5	28-40	2	14-40
2	18-30	0.4	6-23
0.4	8-18	0.18	2-15
0.18	6-14		
0.075	5-10		

In Figure 55 and Figure 56 are reported the average particle size distribution of the three types of granite by-products, these are compared with the two gradings imposed by the C.N.R. 29/72 specification. The average particle size distribution of the three granite by-products is related to the tests conducted on samples deriving from the construction site.

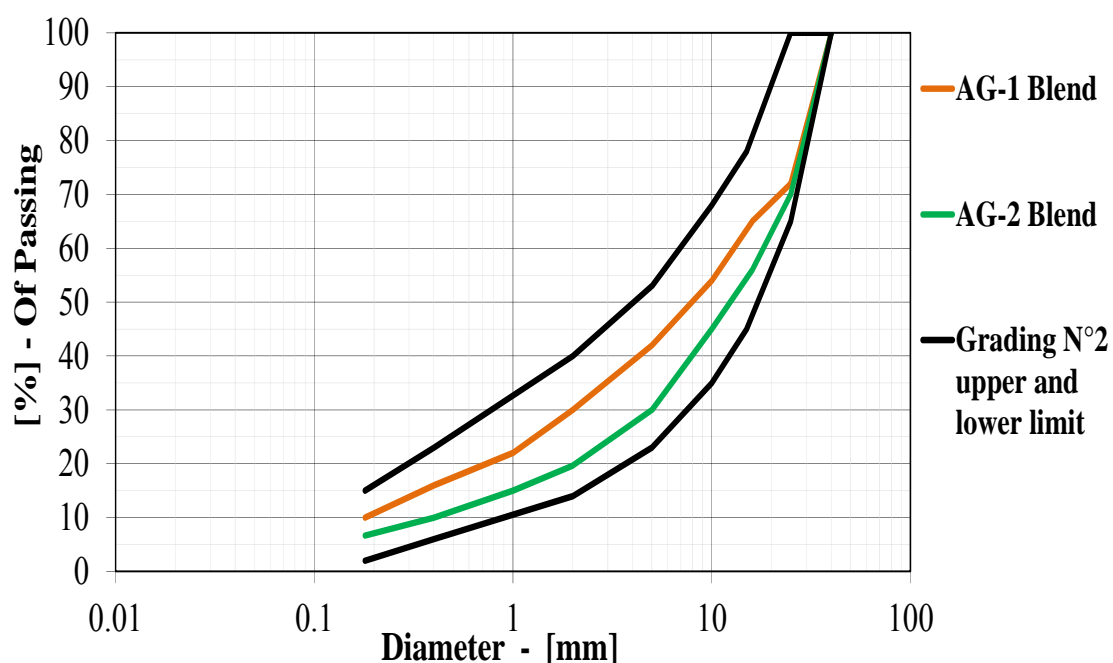


**Figure 55. Cement bound granular mixtures, grading for Major Highways**



**Figure 56. Cement bound granular mixtures, grading for Minor Highways**

The mixtures were obtained using the right percentage of aggregates in order to obtain a blend that will fit inside the grading for minor highways. In Figure 57 are reported the two blends in order that they are inside the grading n°2.



**Figure 57. AG-1 and AG-2 blend in order to be inside the grading for Minor Highways**

Tests were performed preparing samples for each of the two blends. Samples were prepared by mixing the aggregates with the specified water content and Portland cement percentage see Figure 58.



**Figure 58. Mixing the aggregates with water and Portland cement**

Once the mix was ready it was placed inside the mould in five different layers each layer was compacted (Figure 59). The compaction was performed using the same compacter used for the modified proctor test.



**Figure 59. Samples after compaction**

After the compaction procedure was terminated the upper surface of the samples was smoothened in order to have an even surface on which perform the compressive strength test. Samples were then left in the mould to mature for 24 h. After 24 h the samples were removed from the mould (Figure 60) and placed in wet sand in order to mature other 7 days.



**Figure 60. Samples removed from the moulds**



In Figure 61 it possible to see the wet sand in which the samples must stay in order to mature for 7 days.



**Figure 61. Samples maturing for 7 days under wet sand**

After 7 days have passed it is possible to perform compressive strength tests. Specimens are placed in the loading press and tests are performed Figure 62.



**Figure 62. Specimens ready to perform compressive strength test**

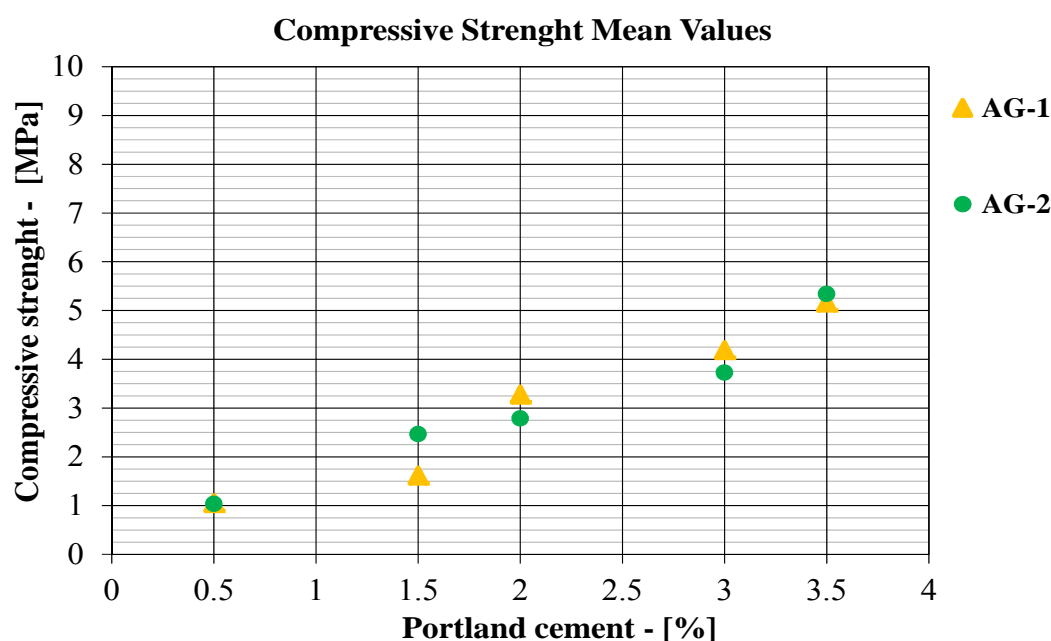


In Figure 63 it is possible to see a specimen after that the compressive strength test has been performed.



**Figure 63. Sample after test**

In Figure 64 are reported the mean values of compressive strengths resistance for the two types of aggregates tested. The C.N.R. 29/72 specification imposes that the compressive strength value must be between 2.45 and 4.41 MPa. These values are obtained for booth aggregates with percentages of cement between 1.5% and 3%.



**Figure 64. Compressive strength mean values**

To better understand the values reported in the chart Figure 64 the values have been reported in Table 14 in which not only cement content and compressive strength values have been reported but also the water content that has been used to be able to prepare the specimens.

**Table 14. Mean Compressive strength values related to cement and water content**

Sample	Cement Content (%)	Water Content (%)	Compressive Strength (MPa)
AG-1	0.50	6.60	1.05
	1.50	6.70	1.62
	<b>2.00</b>	<b>6.80</b>	<b>3.28</b>
	<b>3.00</b>	<b>7.10</b>	<b>4.19</b>
	3.50	7.30	5.16
AG-2	0.50	5.60	1.03
	<b>1.50</b>	<b>5.70</b>	<b>2.46</b>
	<b>2.00</b>	<b>5.90</b>	<b>2.79</b>
	<b>3.00</b>	<b>6.30</b>	<b>3.73</b>
	3.50	6.50	5.34

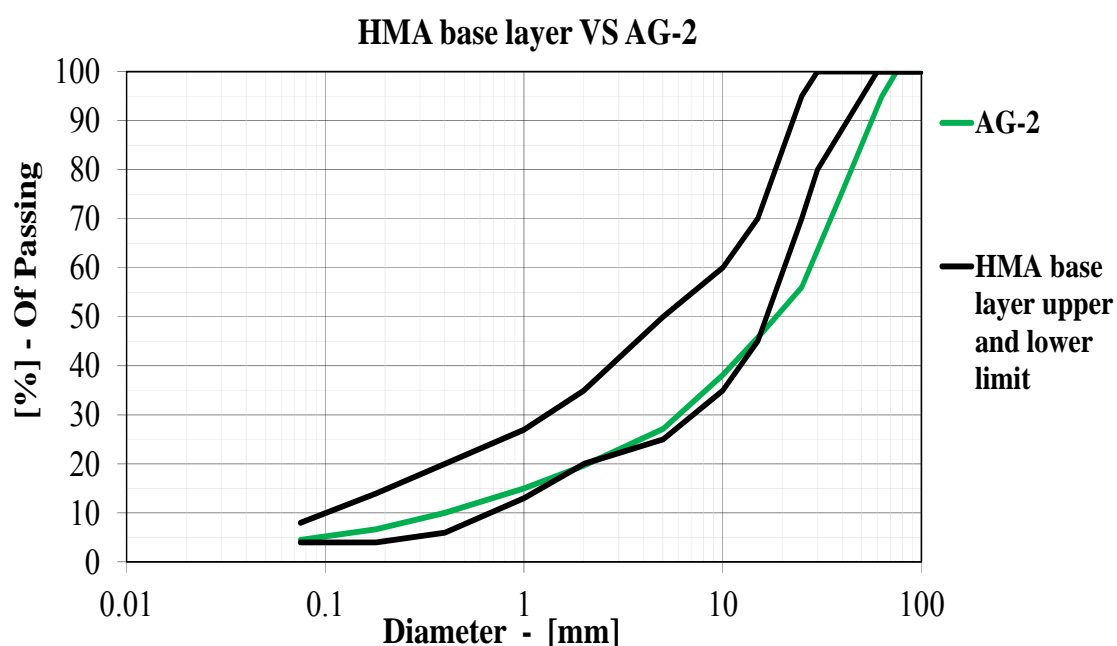
## 6.5 Tests and granite by-products to be used in bitumen bond layers

The last phase of the research was concentrated on using granite by-products to evaluate the possible application in HMA layers. The AG-2 aggregate was chosen because of the three showed better Los Angeles abrasion values. The grading that was used was that of HMA base layers for Minor Highways following the Italian Ministry of Transportation specifications. In Table 15 are reported the upper and lower limits imposed by the specification.

**Table 15. Upper and lower limits for HMA base layer**

Grain Diameter (mm)	HMA base layer
40	100-100
30	80-100
25	70-95
15	45-70
10	35-60
5	25-50
2	20-35
0.4	6-20
0.18	4-14
0.075	4-8

In Figure 65 are reported the upper and lower limits for HMA base layers in comparison with AG-2 average grading. Because AG-2 doesn't fulfill entirely the specified grading it was necessary to prepare a blend with the right percentage of aggregates.



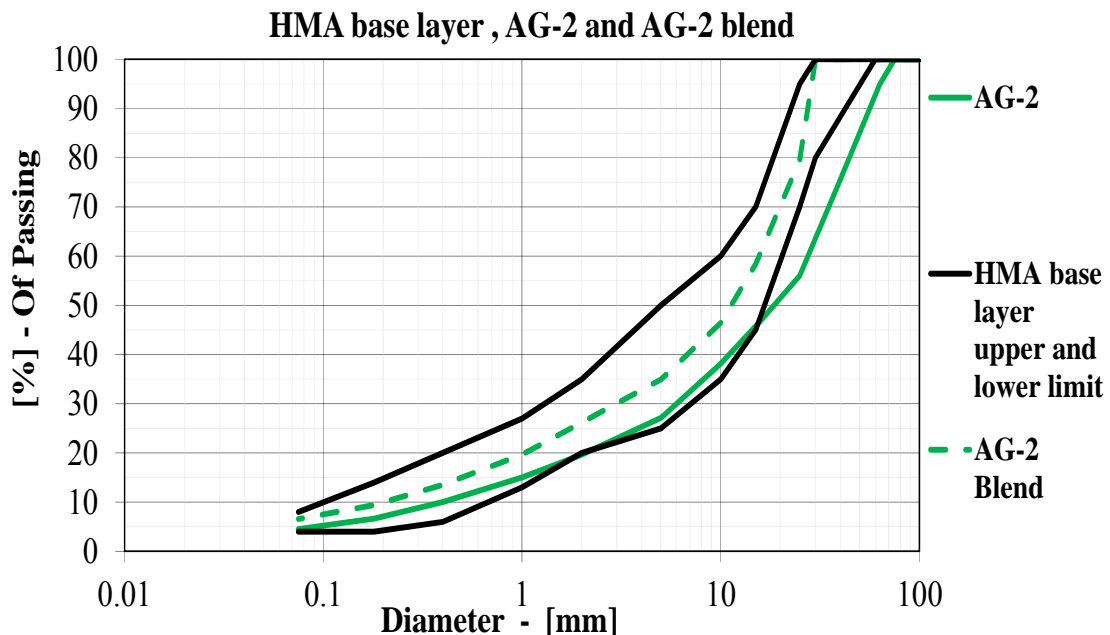
**Figure 65. Comparison between HMA base layer grading and AG-2 average grading**

In Figure 66 it is possible to see the preparation of the blend by mixing each fraction in the right percentage in order to have a blend that can fulfill HMA base layer grading.



**Figure 66. Preparation of each aggregate fraction for the grading of HMA base layers**

In Figure 67 it is possible to see the obtained grading AG-2 blend obtained by mixing the right proportion the aggregates. AG-2 blend is well inside the grading and at this stage it was possible to prepare specimens.



**Figure 67. Comparison between HMA base layer grading, AG-2 average grading and AG-2 blend**

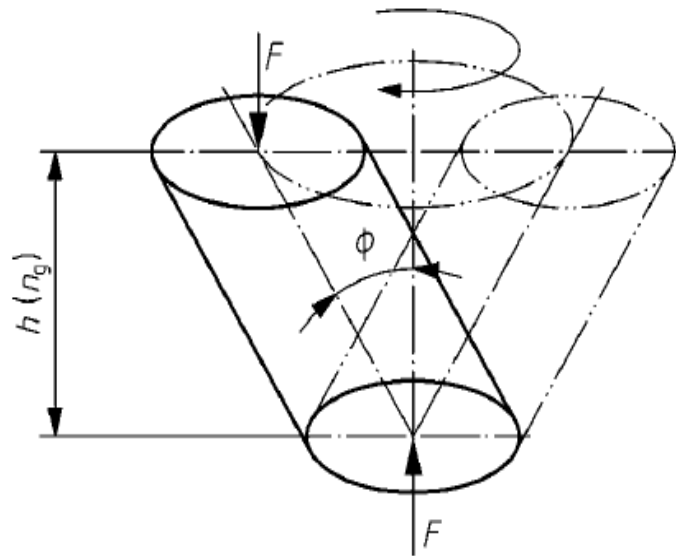
To prepare the HMA specimens the obtained blend was mixed with bitumen type 50/70. To prepare each specimen 5% of bitumen was used expressed as percentages of the total mass of the aggregate, while the total filler was of 3%. Filler was obtained from granite fines, it has been studied that granite fines can have a potential application in asphalt production (Riberio et al., 2008).

Before mixing the two components together aggregates were heated at a temperature of 170°C while bitumen was heated at a temperature of 165 °C.

The method of compaction that was used was that of gyratory compaction according to UNI-EN 12697-31 (UNI-EN 12697-31, 2007) specifications. This compaction method combines a rotary shearing action and a vertical resultant force applied by a mechanical head. The procedure consists on placing the bituminous mixture in a cylindrical mould limited by inserts and kept at a constant temperature within specified tolerances throughout the whole duration of the test.

Compaction is achieved by the simultaneous action of a low static compression, and of the shearing action resulting from the motion of the axis of the mould which generates a conical surface of revolution, while the ends of the test piece should ideally remain perpendicular to the axis of the conical surface.

$F$ : axial resultant force;  $h (n_g)$ : height of specimen after a number of gyrations,  $\phi$ : angle



**Figure 68. Principle on which the gyratory compaction method is based**  
(source UNI-EN 12697-31)

The following parameters for the operation of the compactor are: compaction pressure of 0.6 MPa, an external gyration angle of  $1.25^\circ$  with 30 gyrations per minute. In Figure 69 is shown the gyratory compactor machine used to prepare the specimen. In order to perform the tests additional information's are: aggregate specific gravity, percentage of filler and of bitumen, bitumen type. In this phase a total of five specimens were prepared.



**Figure 69. Gyrocompactor**

In Figure 70 it is possible to see one of the specimens extracted from the mould. The five samples obtained in this phase of the test campaign showed immediately some defects. Aggregates in some areas of the specimen resulted not covered by bitumen and it was possible to see structural failure of the specimens. From a first analysis it seemed like this problems could be attributed to a major presence of fines.



**Figure 70. Extraction of the specimen from the mould**

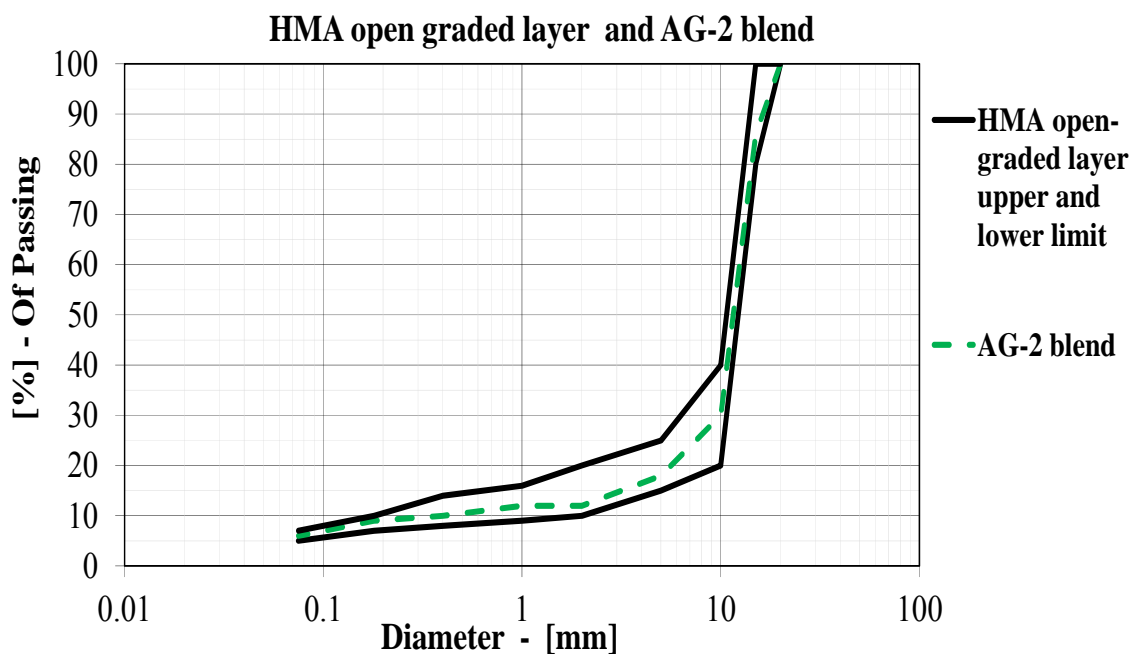
For this reason new samples were prepared with minor arrangements. First of all aggregates fractions from 0.18 mm to 40 mm were washed in order to eliminate any trace of possible fines on the surface, also the amount of filler was reduced to 2% while bitumen remained 5%. Test conducted with the introduction of these variations showed some improvements but more could be done. Thinking that the problem could be the type of filler used obtained by granite, new samples were prepared using as grading for Open-graded HMA layer, in which the presents of filler is marginal or absent. In Table 16 are reported the upper and lower limits of the grading for HMA open-graded layers.

**Table 16. Upper and lower limits for Open-graded HMA layers**

Grain Diameter (mm)	Open-graded HMA layer
20	100
15	80 – 100
10	20 – 40
5	15 – 25
2	10 – 20
0,4	8 – 12
0.18	7 – 10
0.075	5 – 7



In Figure 71 are reported the upper and lower limits of the open-graded HMA layers and the grading obtained by blending different percentages of aggregates of type AG-2.



**Figure 71. HMA open grade layer and the AG-2 blend obtained**

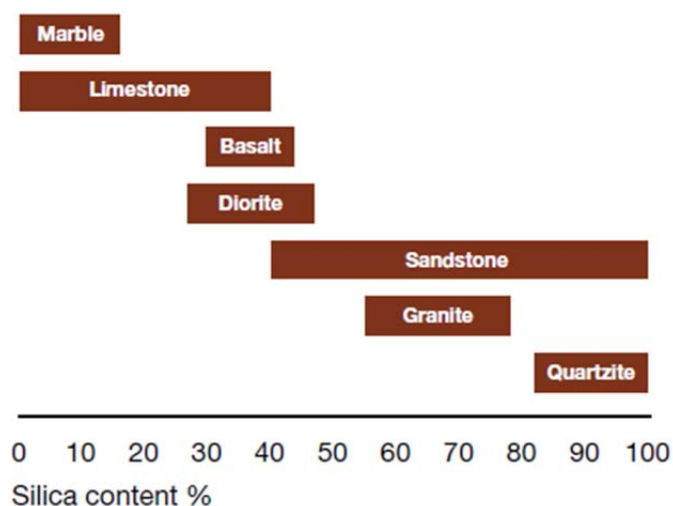
The specimen obtained with this new type of grading (see Figure 72) resulted more uniformly covered of bitumen and now apparent failure could be detected, but still problems appeared. For this reason the preparation of HMA specimens was stopped and the research focused the attention on verifying the possible causes that influenced the cohesion between granite by-products aggregates and bitumen. For this reason stripping tests were performed on such aggregates.



**Figure 72. Open-graded HMA specimen**

## 6.6 Affinity between granite by-products and bitumen

To evaluate the affinity between granite by-products and bitumen specific tests were conducted. It is known that granite has adhesion problems with bitumen. The main factor that is the cause of this problem is related to the nature of these two materials. Bitumen is an oily material and therefore very hydrophobic. This means that bitumen cannot adhere to a wet surface and can be replaced by water over time. In practice the adhesion between bitumen and aggregate depends on the chemical nature of the components and therefore the source of the bitumen and type of aggregate. Other aggregate properties such as surface texture, porosity, shape and absorption will also influence the aggregate/bitumen adhesion. Due to its chemical composition, bitumen has quite a low polarity whereas water is extremely polar. Aggregates may be of an “acidic” type, with surfaces that tend to be negatively charged, or “basic” with surfaces that tend to be positively charged. Acidic aggregates include those with high silica contents like granite, while basic aggregates include carbonates. In Figure 73 is reported the Silica content for the more common aggregates.



**Figure 73. Silica content for some common aggregates**

Following these characteristics it is easy to see that granite aggregates and bitumen have adhesion problems. In order to evaluate the affinity between granite and bitumen stripping tests were performed. Susceptibility to stripping is an indirect measure of the power of a binder to adhere to various aggregates, or of various binders to adhere to a given aggregate.

Tests were performed following C.N.R B.U. 138/92 procedure slightly different to the UNI-EN 12697-11:2012 (UNI-EN 12697-11, 2012) protocol. There are three methods to conduct such test: rolling bottle test, static test method and boiling water stripping test method. In the rolling bottle method, the affinity is expressed by visual registration of the degree of bitumen coverage on un-compacted bitumen-coated mineral aggregate particles after influence of mechanical stirring action in the presence of water. In the static test method, the affinity is expressed by visual registration of the degree of bitumen coverage on un-compacted bitumen-coated mineral aggregate particles after storage in water. While in the boiling water stripping test method, the affinity is expressed by determining the degree of bitumen coverage on un-compacted bitumen-coated aggregate after immersion in boiling water under specified conditions. In this research the static tests method was performed on the three granite by-products plus on basalt aggregates. Basalt was chosen to in order to have comparison between basic aggregates and acid aggregates (granite) and also because is widely used for HMA layers. For each granite by-product two samples were prepared consisting of 0.4 kg that is an average of n°150 particles of aggregate passing the 10 mm sieve and retained on the 6,3 mm sieve. At this stage aggregates were washed and placed in oven at a set temperature of 130°C also the binder and the mixing bowl were placed in the oven at the same temperature of 130°C. The quantity of binder was equivalent to 3.5% by mass of the aggregate particles. Once the aggregates and bitumen have reached the set temperature they are poured in the mixing bowl for mixing procedure. The mixing procedure can be done by hand and must continue until all the aggregates are completely coated. If any particles are not completely coated after five minutes of mixing, new aggregates must be tested repeating the procedure with an increased proportion of binder. The binder shall be increased by steps of 0.5 % by mass of the aggregate particles until a mix giving complete coating of the aggregate is obtained. At this stage we have specimens of un-compacted bitumen-coated aggregate shown in Figure 74. The trays will stand to cool off for 1h and then each sample of coated specimen will be placed in distilled water. For each aggregate one sample was placed at a constant temperature of 25°C and the other at 40°C for 48h.



**Figure 74. Un-compacted coated aggregates**

In order to have constant temperature specimens were placed in a thermostatic bath. In Figure 75 samples placed in a thermostatic bath at a constant temperature of 40°C.



**Figure 75. Specimens at constant temperature of 40°C**

After immersion for the set period, the specimens were extracted from the distilled water and left to dry. To verify the presence of particles with incomplete coating by the binder a visual observation was carried out. In Figure 76 and Figure 77 are shown respectively basalt aggregates and granite by-products deriving from crusher subjected to a constant temperature of 40°C in distilled water. While in Figure 78 and Figure 79 are shown granite by-products deriving from TBM and D&B still subjected to distilled water at 40°C.



**Figure 76. Basalt aggregate**



**Figure 77. Aggregate from crusher**



**Figure 78. Granite from TBM**



**Figure 79. Granite from TBM**

The other set of samples was subjected to a constant temperature of 25°C in distilled water in Figure 80 and in Figure 81 are shown samples deriving from basalt aggregates and granite by-products from crusher. While in Figure 82 and Figure 83 are shown respectively granite by-products deriving from TBM and D&B.





**Figure 80. Basalt Aggregates**



**Figure 81. Granite from crusher**



**Figure 82. Granite from TBM**



**Figure 83. Granite from D&B**

The amount of surface that results uncovered by bitumen after the stripping process are reported in Table 17.

**Table 17. Values obtained by visual observation**

Samples	Temperature 25°C	Temperature 40°C
	Incomplete coating (%)	Incomplete coating (%)
Basalt	2	6
Granite from Crusher	5	38
Granite from TBM	48	70
Granite from D&B	10	45

The percentage of stripping imposed by the Italian Ministry of Transportation for a base layer is  $\leq 5\%$  at a set temperature of 40°C. The results have shown very high values of stripping for granite deriving from TBM, while the other two by-products show lower values and quite similar to each other. It is very interesting to see that although the by-products derive from the same mother rock with same chemical composition, they behave quite differently when in contact with bitumen.

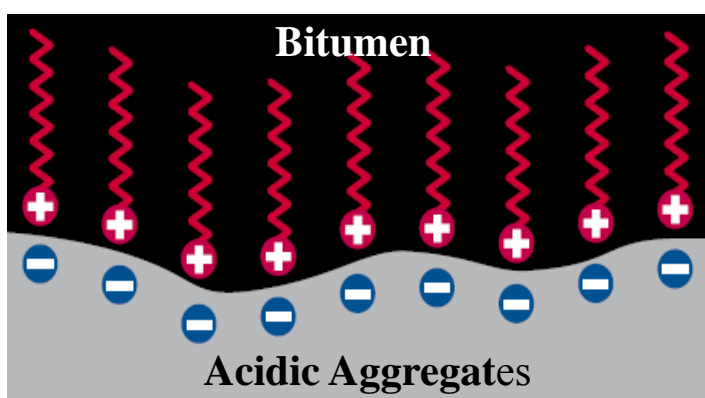
One of the reasons of such behavior can be explained by the kind of surface: rugosity, porosity and other surface characteristics. This can be attributed to the type of process that the granite by-products have been subjected to.

Granite by-products deriving from TBM show smoother surfaces than the other two and are less porous. According to the results obtained granite by products are not suitable for HMA base layers, but further investigations are needed. To evaluate the possibility of increasing adhesion stripping tests were performed once more but this time adhesion promoters were used in the mix and stripping was evaluated.



## 6.7 The use of adhesion promoters to increase bonding between bitumen and granite aggregates

Adhesion promoters are cationic surface active molecules which concentrate at the bitumen aggregate interface. While the positive hydrophilic head groups on the surface active agents bind strongly to the negative sites on the aggregate surface, the hydrophobic hydrocarbon "tails" of the molecules anchor into the bitumen. The adhesion promoter thus acts as a bridge or glue between the bitumen and the aggregate surface which resists the displacing effect of water. In Figure 84 is reported how active adhesion promoters work when introduced into a system with bitumen and acidic aggregates.



**Figure 84. Adhesion promoters function between bitumen and acidic aggregates**

Tests to evaluate the power of adhesion promoters were conducted performing the UNI-EN 12697-11:2012 test. This time the boiling water test was performed. This test consists on measuring the affinity expressed by determining the degree of bitumen coverage on uncompacted bitumen-coated aggregate after immersion in boiling water under specified conditions. The aggregate used were those that showed the worsts adhesion parameter for this reason granite by-products deriving from TBM were tested. The aggregate fraction this time was used passing the 10 mm sieve and retained at the 6.5 mm sieve. The quantity of binder was equivalent to 2.1% by mass of the aggregate particles. Three samples were prepared one with no adhesion promoter and the other two with two different adhesion promoters produced by Iterchimica the amount was equivalent to 0.3% by mass of bitumen.

To perform the test the mixtures were prepared and then were left to cool off before placing the samples in boiling water for ten minutes. And then evaluate the stripping of each of the samples.

In Figure 85 is shown the granite by-product deriving from TBM with no adhesion promoter in the mixture. It is possible to see that that all the particles are nearly completely uncovered by the binder.



**Figure 85. Granite by-products with no adhesion promoter**

In Figure 86 are shown aggregates in which the adhesion promoter has been used and it is possible to see that stripping of the binder still is visible but has been reduced if compared with Figure 85.



**Figure 86. Granite by-product with adhesion promoter (Iterlene IN 400-S)**

In Figure 87 are shown aggregates in which adhesion promoters have been used in the mixture it is possible to see that the result obtained is encouraging.



**Figure 87. Granite by-product with adhesion promoter (Iterlene PE 31-F )**

In Table 18 are reported the stripping values respectively for granite by-products with no adhesion promoters, with adhesion promoter type Iterlene IN 400 S and adhesion promoter type Iterlene PE 31-F. It is possible to see how the use of adhesion promoters has drastically reduced the stripping of bitumen from granite particles. The possible application of adhesion promoter Iterlene PE 31-F in percentage of 0.5% by mass of bitumen could probably reduce the stripping in order to reach the 5% value imposed by Italian legislations to use such aggregates in HMA base layers.

**Table 18. Stripping values for the three tested samples**

Samples	No adhesion promoter	Adhesion promoter (Iterlene IN 400-S)	Adhesion promoter (Iterlene PE 31-F)
Granite from TBM; 2.1% of bitumen (35/50)	90 %	40 %	15 %

## **7. DIGITAL IMAGE PROCESSING**

### **7.1 Introduction to image processing**

Image processing techniques have as their objective the extraction of information from digital images. The image can be acquired using several different devices depending on the case of study and also the scale of resolution considering macroscopic or microscopic scale. The techniques of image processing and computer vision are now commonly used in many scientific fields, ranging from the medical diagnostics to the industry in order to control process, also used in microbiology and meteorology.

The study developed in this phase of the research was thought in order to introduce this technique to determine the possibility of using digital image processing to evaluate in a more objective and rapid way the debonding phenomena in asphalt mixes that can be acquired during stripping tests.

Imaging processing can be divided into two different methods based on the image acquisition technique: static or dynamic. In static image acquisition, the sample is stationary while the images are being captured, whereas in dynamic image acquisition the sample is moving while being captured. In this research static image processing analysis was introduced consisting of the manipulation and analysis of information that can be obtained analyzing the image.

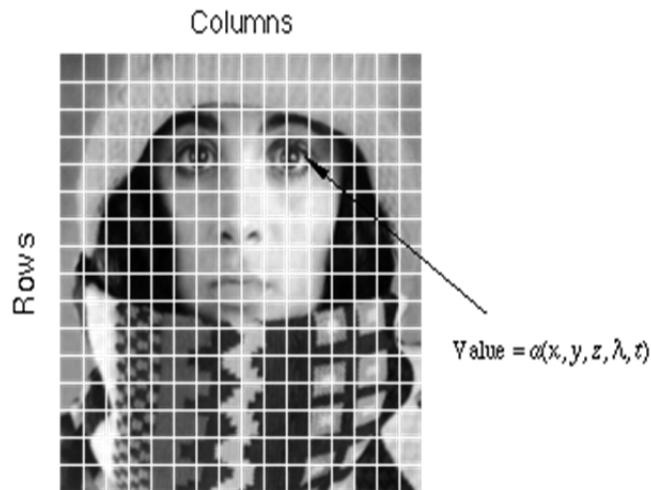
To be able to perform image processing analysis the basic instrumentation needed consists of a computer, provided with an input device capable of acquiring images generally a digital camera attached to a card that decodes these images in digital form, interpreted and displayed by the computer, but also most important a software capable of analyzing the information's that are in the image.

Before introducing the various steps that characterized this phase of the research it is essential an introduction in digital image.

A digital image is a numeric representation described in a 2D discrete space derived from an analog image  $(x, y)$  in a 2D continuous space through a sampling process, which is often referred to as digitization. The effect of digitization is shown in Figure 88. The 2D image  $(x, y)$  is divided into  $N$  rows and  $M$  columns. The intersection of a row and a column is called a pixel. The term "pixel" stands for "Picture Element", is the smallest

among the items that are displayed on a screen, the screen is divided up into a matrix of thousands or even millions of pixels.

The value given to the coordinates  $[m, n]$  are  $m = (0, 1, 2, \dots, M - 1)$  and  $n = (0, 1, 2, \dots, N - 1)$  is a  $[m, n]$ . Other function of variables that affects the face of 2D is depth ( $z$ ), color ( $\lambda$ ) and time ( $t$ ).



**Figure 88. Digitalization of a continuous image**

The image shown in Figure 88 has been divided into  $N = 16$  rows and  $M = 16$  columns. The value assigned to each pixel is the average brightness in the pixel rounded to the nearest integer value. The process of representing the amplitude of the 2D signal as an integer value in a given coordinate with  $L$  different gray levels, it is usually referred to as amplitude quantization or simply quantization.

There are default values for the various parameters encountered in digital image processing. These values can be derived from video models from algorithmic requirements, or the desire to maintain simple digital circuits. Table 19 gives some values commonly encountered.

**Table 19. Referable parameters**

Parameters	Symbols	Typical values
Row	$N$	256, 512, 525, 625, 1024, 1035
Column	$M$	256, 512, 768, 1024, 1320
Levels of grey	$L$	2, 64, 256, 1024, 4096, 16384

In a binary image, there are only two gray levels, which can be defined, for example, as "black" and "white" or "0" and "1".

### **7.1.1 Image acquisition**

The image is acquired in digital form and in different interchange formats: the characteristic elements are the scanning resolution or display and type (B/W, 16 or 256 gray scale or color, millions colors), which identifies the amount of information associated with each pixel (for example, 8 bits per pixel corresponds to 256 colors). The pixel or base unit in an image is identified by its relative position (x, y) and its characteristics of color/intensity expressed as RGB or Gray Level.

The color images, in particular, are represented by the primary colors Red (red), Green (green) and Blue (blue), called RGB color coordinates. In conventional RGB format, therefore, an image is represented, in each considered pixel, as the intensity of these three basic colors. When all three colors are zero, the object's color is black, and when all three colors have the maximum value, the object's color is white. Equal levels of R, G and B generate the gray and then the image is defined by the Gray Level. When we take a photograph of the digital camera performs, in rapid succession a series of steps. First of all, you set the aperture to the value chosen. Subsequently, the shutter opens and let's light in order to impress the digital sensor. The digital sensor converts the light information into electrical impulses and sends them to the circuits devoted to image processing. Here the data will be properly handled and finally saved as a digital file. A digital image is a representation of two-dimensional image through a series of numerical values, which describe depending on the technique used.

The digital image processing is a discipline that involves the use of algorithms to modify a digital image. These algorithms operate on the pixels that make up the image and applying numerical transformation, return a modified image, or a numeric or tabular representative of a particular characteristic of the input image. With the development of digital images processing it is possible to perform complex operations in a significantly very short time. The processing is done on the assumption that raster images are stored as an array of pixels, each of which contains the color and brightness of the point. Programs can change the pixel values to change the image, using different models of algorithms. Usually it

comes to evaluating a group of adjacent pixels and, based on these; determine the value of color and brightness.

## 7.2 Possible use of image processing to evaluate stripping test values

As reported in previous chapters the determination of affinity between aggregate and bitumen can be determined by performing the stripping test. This test consists of determining the percentage of stripping that is the average proportion of the surface area of the aggregate particle from which the binder is soaked off due to the action of water, expressed in a percentage (EN 12697-11:2012). While performing such tests the possibility of using image analysis to detect the portion of surface area of the aggregate in which the binder soaked off was taken into consideration. The following chapter will take into consideration the various steps conducted in order to evaluate the possibility of using static image analysis to be a valid support when performing stripping tests.

The major steps that are used when performing digital image analysis are: image acquisition and enhancement, object phase detection and measurements see Figure 89.

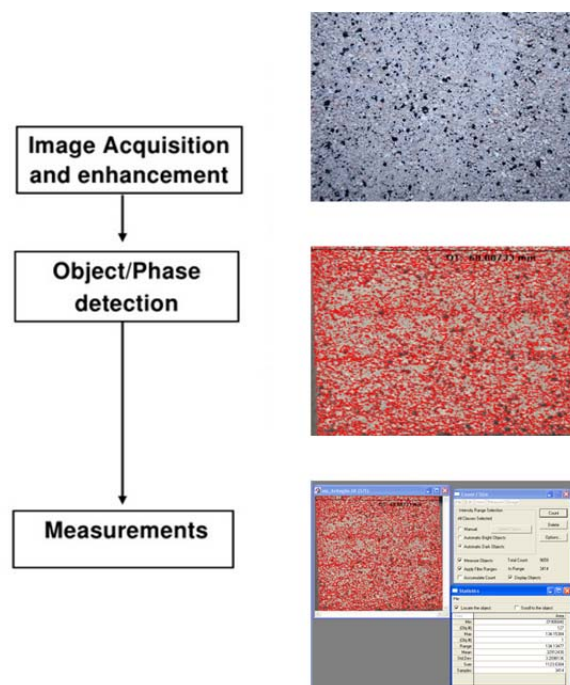


Figure 89. Image analysis major steps of processing

- Image Acquisition: this is the first step or process of digital image processing. Generally, the image acquisition stage involves preprocessing, such as scaling.
- Image Enhancement: the enhancement technique is that of bringing out details that are obscured, or to highlight certain features of interest in an image. Such as changing brightness&contrast.
- Object phase detection: is that of detecting and defining the objects in order to be able to analyze them without considering other elements that are present in the image.
- Measurements: consists in determine and measuring parameters that can involve not only geometrical features but also parameter like colors.

Also it is important to verify if the software used to process the information's that are present in the digital image is capable to measure them, for this reason calibration is needed.

For this reason in the first phase several software's were tested and compared to evaluate if they had the requirements and the potential to be used for the purpose that we wanted. Among all the software tested two of these were chosen and a preliminary calibration was conducted. The research continued using only the best suitable software of the two.

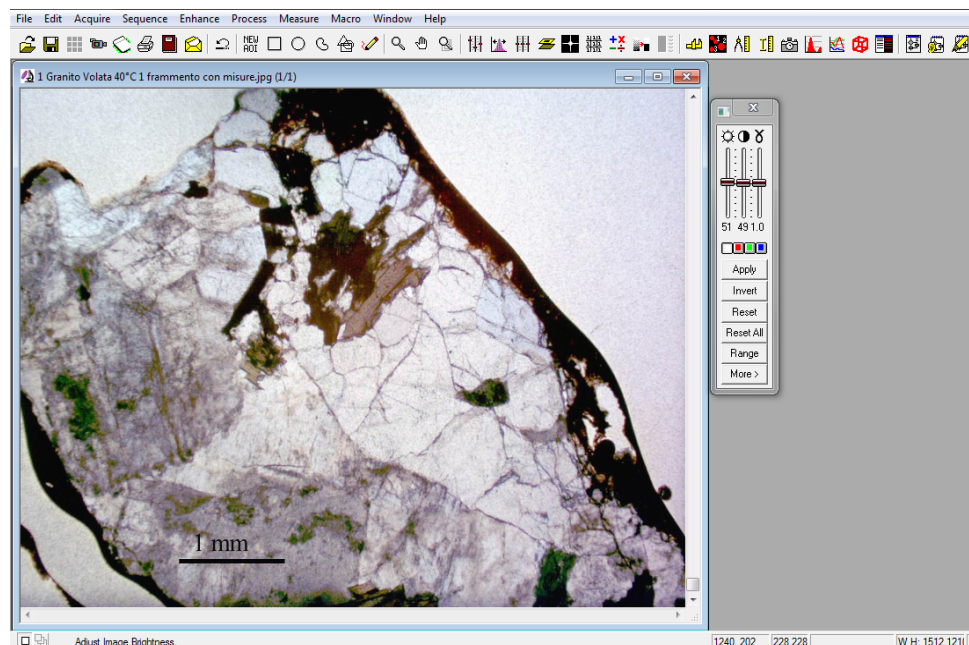


### 7.2.1 Software image processing features

The software's chosen to conduct the first phase of the research were two: Image-Pro Plus version 6.0 and Pixcavator. The main features of each of the used software's will be explained.

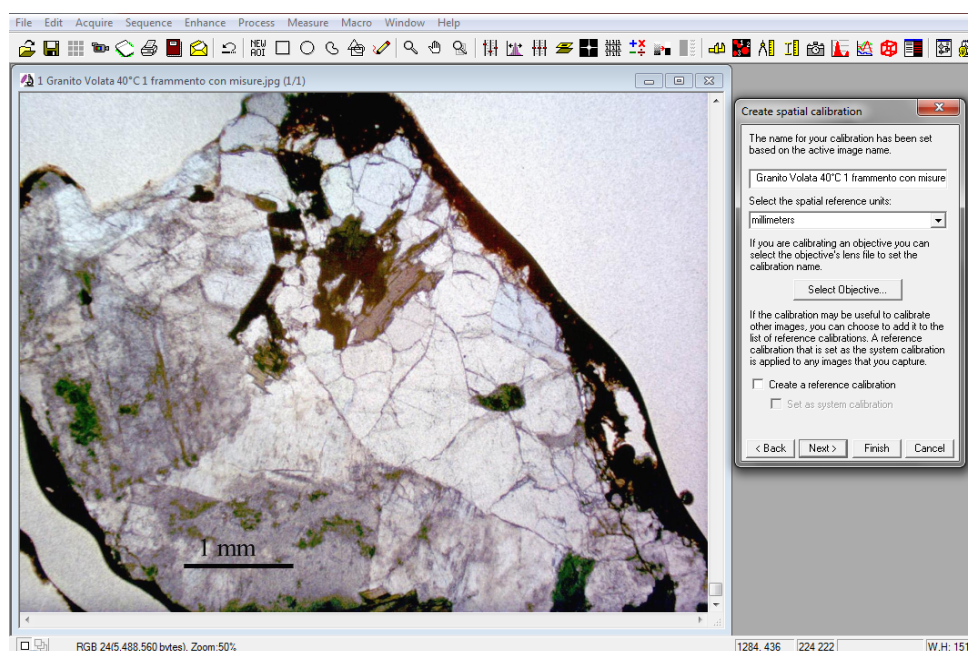
Image-Pro Plus version 6.0 is very versatile software with many useful tools. Once an image is acquired it is always necessary some form of enhancement to improve its appearance for aesthetic reasons and to improve the ability to extract data from it. Enhancement techniques range from simple operations such as brightness, contrast adjustment and gamma corrections to the sophisticated and complex spatial and morphological filtering operations designed to “tease out” and refine visual information.

Brightness is a term used to describe the overall amount of light in an image. In Image-Pro Plus, brightness is modified using the “Brightness” slider controller on the BCG controls. Contrast is a term used to denote the degree of difference between the brightest and darkest components in an image. Gamma correction is a specialized form of contrast enhancement that is designed to enhance contrast in the very dark or very light areas of an image. All these tools are shown in Figure 90.



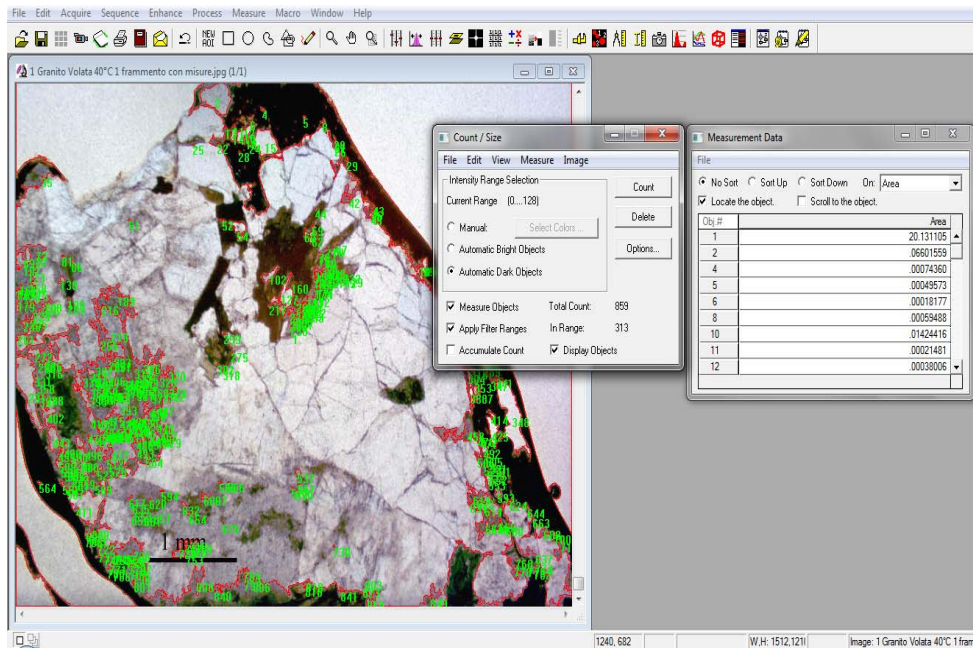
**Figure 90. Image Pro Plus brightness, contrast and gamma dialog box.**

In addition to contrast adjustment, a wide variety of specialized filters and operators can provide further image enhancement. These include a wide variety of edge and detail enhancement strategies, ways of separating structures through erosion and segmentation so that they can be counted and methods for subtracting uneven backgrounds from images. Before taking any measurements from the image it is important to calibrate it. The button allows the correct calibration to be selected for the image's scale or magnification. The calibration is set via the dialogue at Measure/Calibrate/Spatial. Image-Pro Plus's pixel-level measurements can be scaled to fit any coordinate system. This allows obtaining measurements which are reported in terms meaningful to the application. For example, it is possible to calibrate the measurement scale to meters or centimeters or any measuring scale. Image-Pro Plus will express your measurements in terms of that unit. Additionally, if your image contains a measurable object of the unit length, you can calibrate your scale directly from that object, using the Image button in the Spatial Calibration dialog box Figure 91.



**Figure 91. Calibration dialogue box**

**Count and Measure** allows automated gathering of data with specified parameters and **Manual Measurement** allows individual manual measurements to be made. In Figure 92 is shown the count and size dialogue box together with the measuring results.



**Figure 92. Count and measure dialogue box**

These are only some of the main tools that this software is capable of performing more features will be explained in the next chapters.

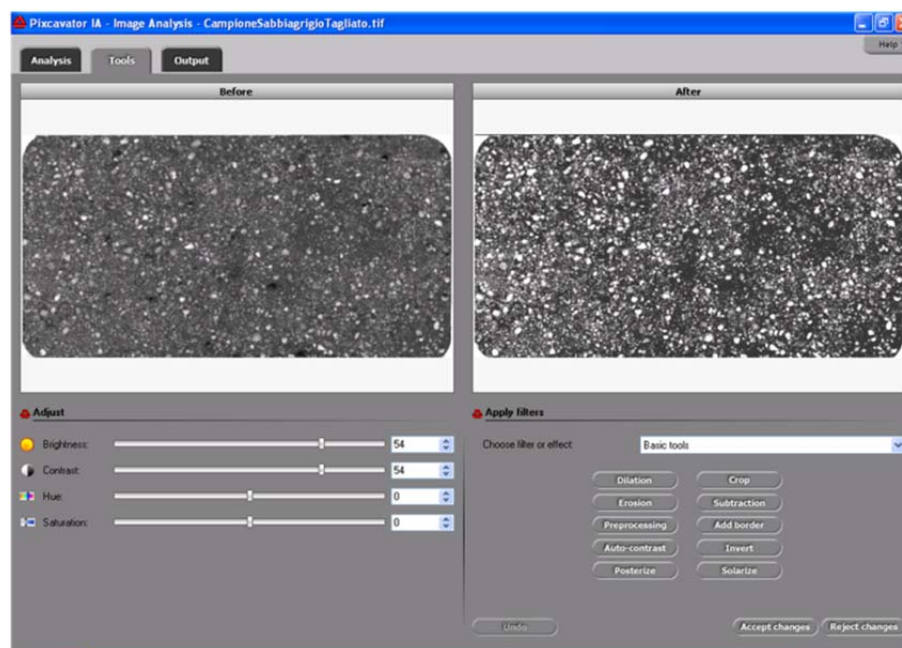
Image analysis with Pixcavator includes three phases:

- **Step Auto**: creates a data structure that contains information about all possible objects in the image.
- **Semi-automatic**: the operator can change the characteristics of the objects according to the requirement. These changes are immediate and the output data are updated in real time.
- **Manual**: the operator can exclude the residual noise and irrelevant details from the analysis by simply clicking on them. Currently, Pixcavator has three processing modulus.
- The module of analysis allows the operator to load the image, and if necessary to reduce the file size of the image. This last option is useful in the case in which the analysis of the image is too "heavy" compared to the hardware used.
- The Tools tab allows the operator to manipulate images using standard image processing operators and any special effects.

- The Output tab allows viewing, navigating and save the data collected and analyzed image with the appropriate changes.

For color images, it is possible to decide which channel you want to analyze. There are three color channels, namely red, green and blue. The analysis of large images will obviously require more processing time. In some cases the smaller details within the image can often be relatively low interest; in this case it is possible to reduce the image. The work begins by opening the tab "Tools". The monitor displays two images, one left and one right. The image on the left side is the original and the one on the right shows the effects of processing taking place. Using the sliders to the left it is possible to edit the original image by adjusting the brightness, contrast, tone and saturation Figure 93.

On the right, there is a menu in which you can select filters and effects. The most important tools for the analysis are the commands of erosion and dilation. These can improve the accuracy of analysis, for example by eliminating any imperfections. Some other features are preprocessing, (combining dilation and erosion) and the auto contrast.

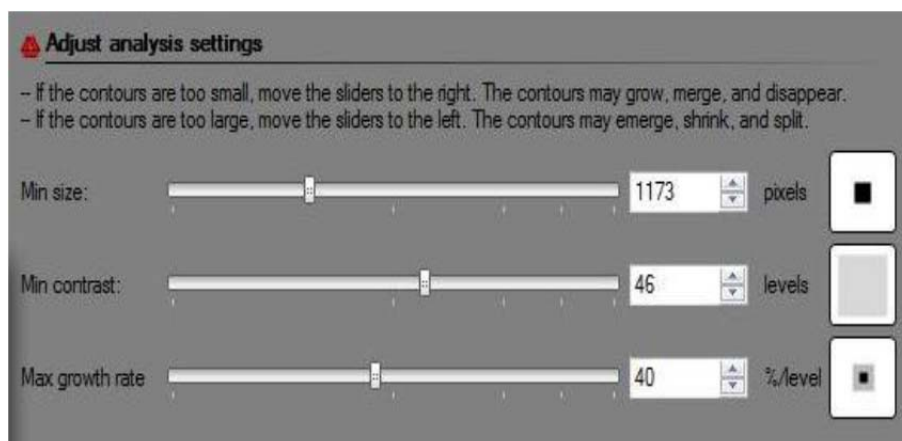


**Figure 93. View of Pixcavator commands**

During processing, a message appears that in the event of an error allow the operator to cancel the command that generated it. When it is considered that the image is ready, it is possible to return to the panel of analysis, and start the phase of processing. At the end of this phase, Pixcavator automatically opens the output card (output). The Output tab is



characterized by multiple options. The results of the automatic phase may or may not be satisfactory. To improve the results semi-automatic mode can be chosen. There are three modules by which settings can be adjusted. This allows to rapidly re-analyzing the image with the new settings Figure 94.



**Figure 94. Main features**

Size and contrast correspond to two key measures of objects in an image to grayscale. The objects that fall below these thresholds will be ignored during the analysis procedure. First, it is necessary to choose the minimum size. For example, if a size of 100 is chosen only items that contain 100 or more pixels are captured. Secondly the value of minimum contrast is chosen. For example, if the value of 20 is chosen, only the objects surrounded by an area with a gray level, which differs from that of the object 20 or more will be captured. The image displayed on the Output tab is updated in real time.

To verify or show better detail, new portion of interest can be added. Positioning the mouse cursor within the image and holding down the Ctrl key, the object closest to the cursor will be marked, and immediately see the measured parameters ( such as its location , its area , perimeter, axes. .. ). In this way it is possible to confirm or exclude arbitrarily that object in the analysis. This is executable over plots in the table which displays the numerical values detected.

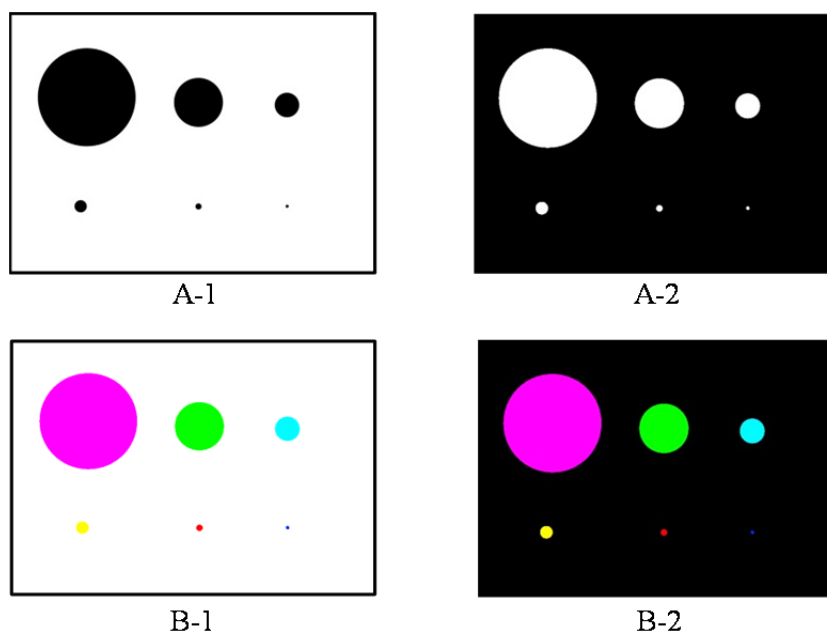
For each object, the data is displayed by the following characteristics:

Type: light or dark;

- Location of the center of gravity;
- Size (area in pixels);
- Perimeter;
- Softness;
- Gray (intensity);
- Contrast medium;
- Dimensions (major and minor axis of the object);

### ***7.2.2 Analysis on images obtained using CAD software***

To perform calibration the phases were two: analyze images reproduced by software using CAD and in the second phase analyze real images. The reconstruction of images using CAD software had the advantage of recreating figures of a known dimension and located in a precise position. Circles of different diameters ranging from 80 mm to 2.5 mm were preliminary reproduced using two types of different backgrounds white or black and different types of colored circles see Figure 95 the resolution of the images was of 842×595 pixel. This was thought in order to evaluate if different color backgrounds and different colored circles will influence the image analysis.



**Figure 95. Images designed using cad software**

In Table 20 are reported the errors made by the two software's when measuring the diameter of the circles. The analysis was first performed on images with black circles and white background (Figure 95 A-1).

It is possible to see how Image-Pro Plus performs better measurements with minor error compared with Pixcavator.

**Table 20. Blacks circles and white background**

Diameter (mm)	Pixcavator error (%)	Image-Pro Plus error (%)
80	0.61	0.09
40	1.13	0.05
20	1.91	0.04
10	3.55	0.08
5	7.54	1.44
2.5	17.15	4.75

Same measurements were performed on white circles and black background (Figure 95 A-2). It was possible to see that the errors made by the two software's were mostly the same of the previous analysis Table 21. It was possible to evaluate on this preliminary tests that the variation of the background from white to black and the variation of the color of the circles did not influence the measurements of the software's.

**Table 21. Errors white circles and black background**

Diameter (mm)	Pixcavator error (%)	Image-Pro Plus error (%)
80	0.60	0.09
40	1.12	0.05
20	1.93	0.04
10	3.56	0.08
5	7.52	1.44
2.5	17.12	4.75

Also tests were performed on images on white and black background but with different coloured circles Figure 95 B-1 and Figure 95 B-2. The errors performed by the softwares are reported in Table 22 for white background it is possible to see that there is a slight difference comparing this values and the values reported for the previous tests.

**Table 22. Coloured circles white background**

Diameter (mm)	Pixcavator error (%)	Image-Pro Plus error (%)
80	0.61	0.08
40	1.13	0.04
20	1.91	0.04
10	3.55	0.08
5	7.54	1.49
2.5	17.15	4.79

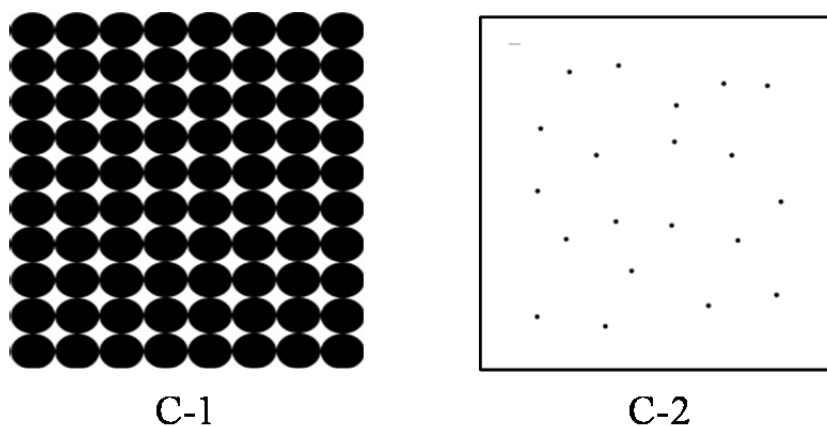
Same measurements were performed on images with black background and colored circles Figure 95 B-2 results are reported in Table 23. It is possible to see that variation of background and the color of the circles do not influence the measurements of the software's.

**Table 23. Coloured circles black background**

Diameter (mm)	Pixcavator error (%)	Image-Pro Plus error (%)
80	0.61	0.08
40	1.13	0.04
20	1.91	0.04
10	3.55	0.08
5	7.55	1.49
2.5	17.16	4.79

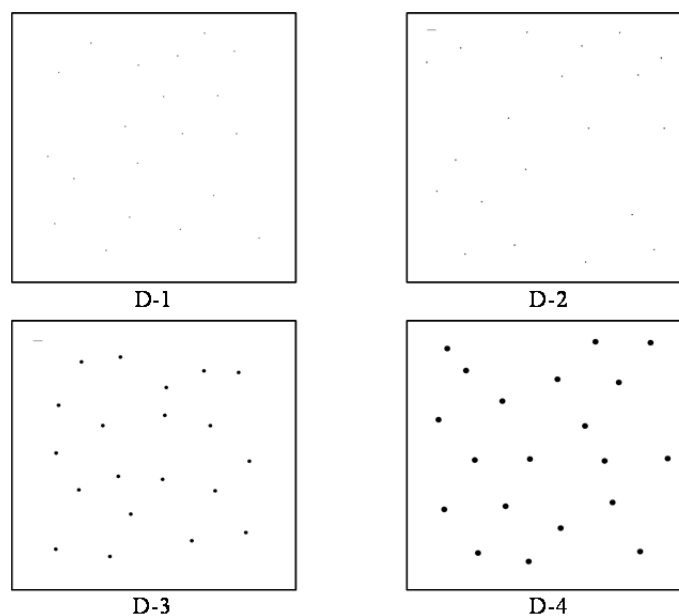


Test continued on measuring multiple objects first of all using circles very close to each other and then multiple circles of the same dimension but randomly positioned.



**Figure 96. Multiple circles**

Due to the position of the circles in which each circle was attached to the one next to him Figure 96 C-1 the two softwares were not able to capture the single diameter of the circles. While in Figure 96 C-2 in which the circles were randomly distributed and not influenced by each other the software could track and measure quite easily the objects. For this reason the research continued on performing tests on randomly distributed circles. Each image had 20 circles each of the same size for a total of five images in which the diameters of the circles ranged from: 5, 3, 1, 0.5 and 0.7 mm. Example of such images are reported in Figure 97. For this set of measurements the resolution of the images was increased passing from 842\*595 pixels to 1512\*1512 pixels.



**Figure 97. Image different diameters**

In Table 24 are reported the mean values of the errors and the standard deviation on the tests conducted using Pixcavator on 5 different measures of diameters. It is possible to see that the errors made by Pixcavator are very large.

**Table 24. Mean values obtained**

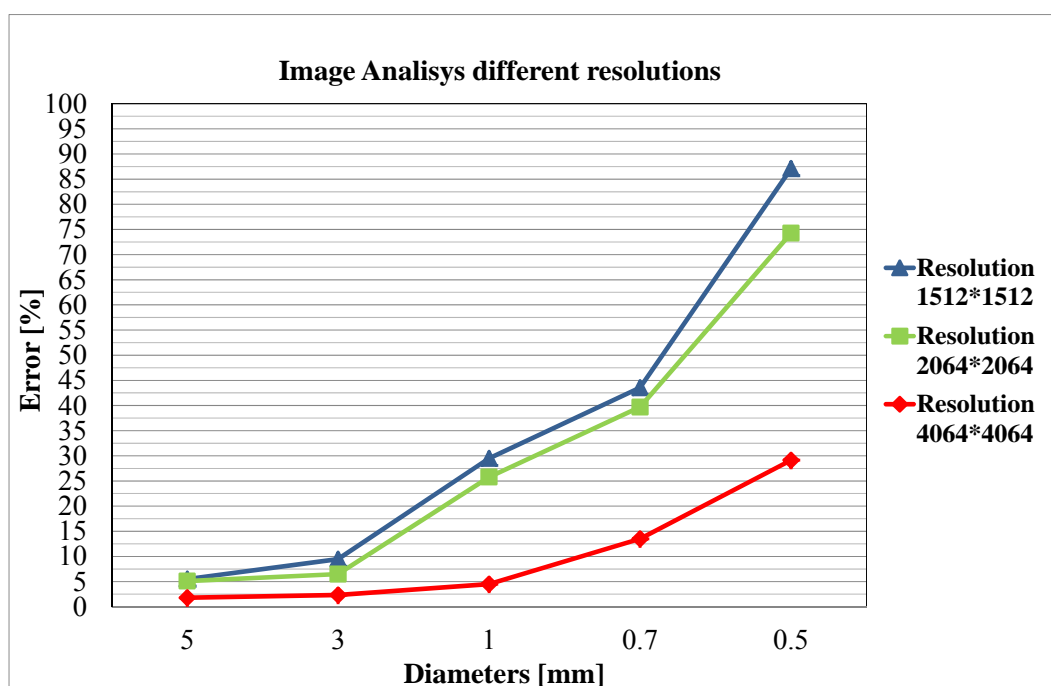
Diameter (mm)	Pixcavator error (%)	Standard deviation (%)
5	8.78	0.22
3	22.33	0.96
1	44.51	2.01
0.7	61.09	4.07
0.5	87.57	4.52

Same measurements were conducted using Image-Pro Plus the values are reported in Table 25. It is possible to see that Image-Pro Plus does better measurements of Pixcavator. From now on all the tests will be performed using Image-Pro Plus software.

**Table 25. Mean values obtained**

Diameter (mm)	Image-Pro Plus (%)	Standard deviation (%)
5	5.49	0.30
3	9.46	0.99
1	29.49	2.12
0.7	43.53	3.83
0.5	87.10	4.78

In order to evaluate in which way the increase of resolution of the images will fetch benefits in terms of precision of the measurements tests were performed using three different types of resolution: 1512\*1512, 2064\*2064 and 4064\*4064 pixels. The results are reported in the chart in Figure 98.

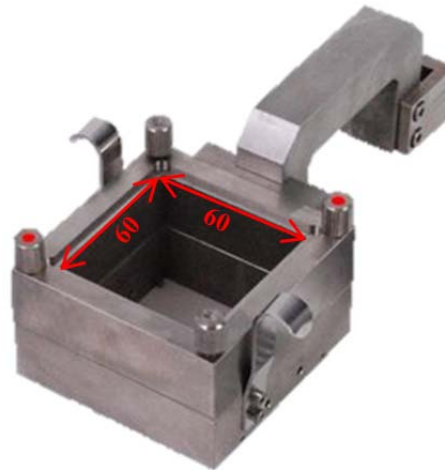
**Figure 98. Image analysis using Image-Pro Plus with images at different resolutions**

It is possible to see with the increase of resolution the decrease of the error. The drawbacks are that you have larger files to deal with and this in a minor way this can influence the time of processing the image analysis.

Having obtained useful informations on the errors that Image-Pro Plus can make on image reproduced by CAD software's the research continued taking pictures of real images by using a digital camera.

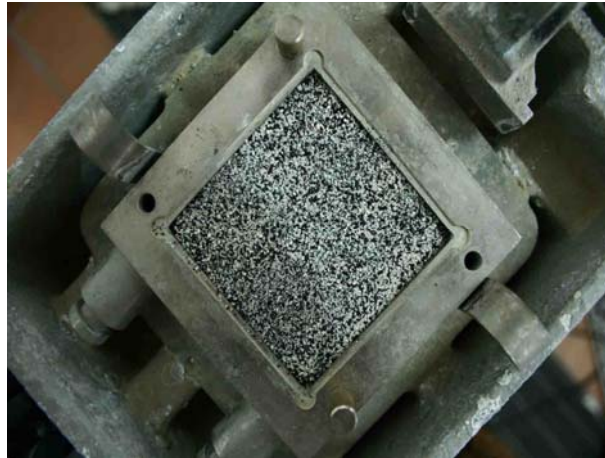
### **7.2.3 Image analysis on real objects**

From the results obtained on the analysis on images reproduced by CAD softwares the study continued in order to evaluate the possible use of the digital image analysis on images obtained by taking pictures of real objects. In this case the scope of our study wasn't that of measuring the dimension of a certain object but that of evaluating the percentage of a certain region covered by two types of colors. Two different types of material were used. Two calibrated materials were used: white sand of known diameter (1 mm) and copper slag of black colour also calibrated and had the same diameter of the sand. The images analysed in this research were captured using a Canon 1100 D digital still camera, which has an image resolution of 12.2 Megapixels. Also to conduct the experiments a sample older was needed, a shear box sample older was used with the following dimensions 60\*60 mm the exact dimension of the sample holder was important wen scaling the image from pixels to mm Figure 99.



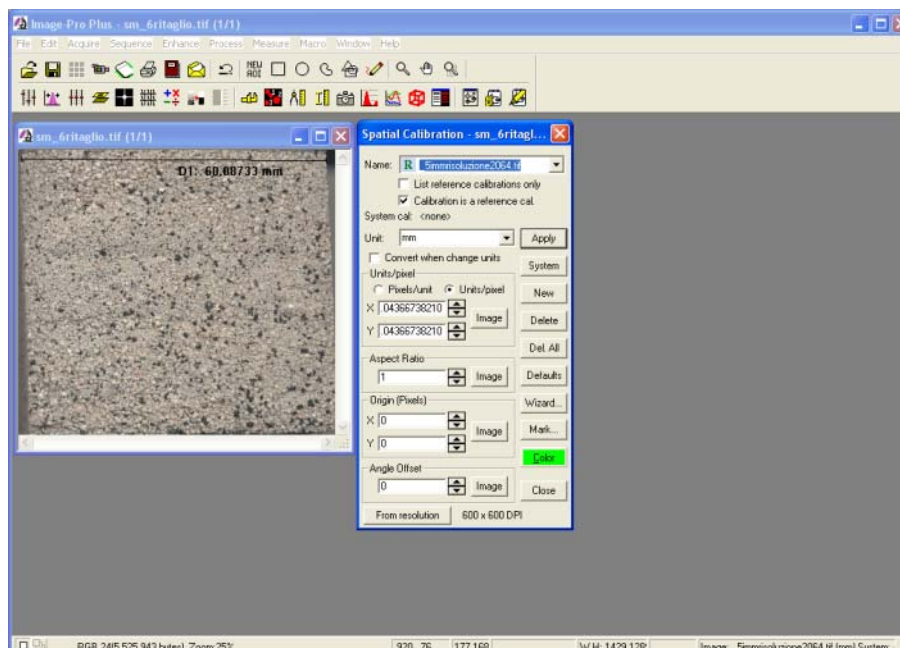
**Figure 99. Sample holder**

In order to perform the tests, sample were prepared by mixing different percentages of sand and copper slag and introduced in the sample holder Figure 100.



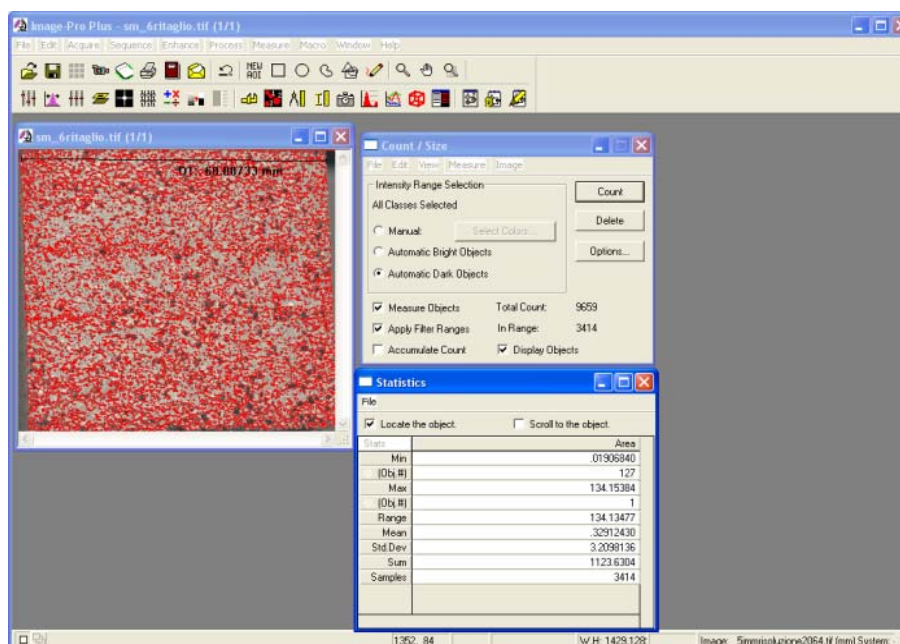
**Figure 100. Sample ready for image analysis**

The methodology was divided in three phase's preparation of the sample, image capture using the camera and digital image processing using the software. Each phase was obtained in order to have a process that could be repeated several times. The mixes were prepared each time with different percentage in weight of sand and copper slag. The sample was introduced inside the sample holder and images were taken. Once the images were saved in the data base, the software was used to perform digital image processing. First of all calibration was performed by using the dimensions of the sample holder and also knowing the dimension of the calibrated sand but also the resolution of the image that had to be analyzed. In Figure 101 is shown a phase of calibration using Image-Pro Plus software. Soon after calibration special filters were used in order to better track the different color objects.



**Figure 101. Software image calibration**

Soon after calibration special filters were used in order to better track the different color objects. In Figure 102 is shown one of the phases of the tracking of black objects in this search the software is measuring the area of the black objects.



**Figure 102. Object search**

The scope of this phase of the research is that of evaluating the error made during all these processes when evaluating the percentage of white and black colors reported to the entire area. In Table 26 are reported the values of the percentage of error made analyzing different samples prepared with different percentages of sand and copper slag.

**Table 26. Error made during the image analysis**

Number of sample	Percentage sand (%)	Percentage copper slag (%)	Error in the measurements (%)
1	90	10	1
2	80	20	2
3	10	90	1
4	30	70	1.5
5	50	50	2.5
6	40	60	2
7	60	40	1
8	10	90	2

The data obtained from this set of tests were useful to be able to evaluate the maximum error when using this type of analysis to search two different types of colors that present in a picture the maximum error made was of 2.5 % and this was considered an exactable value for the type of application that we want to use this method for.

### 7.3 Digital image processing to evaluate stripping test values

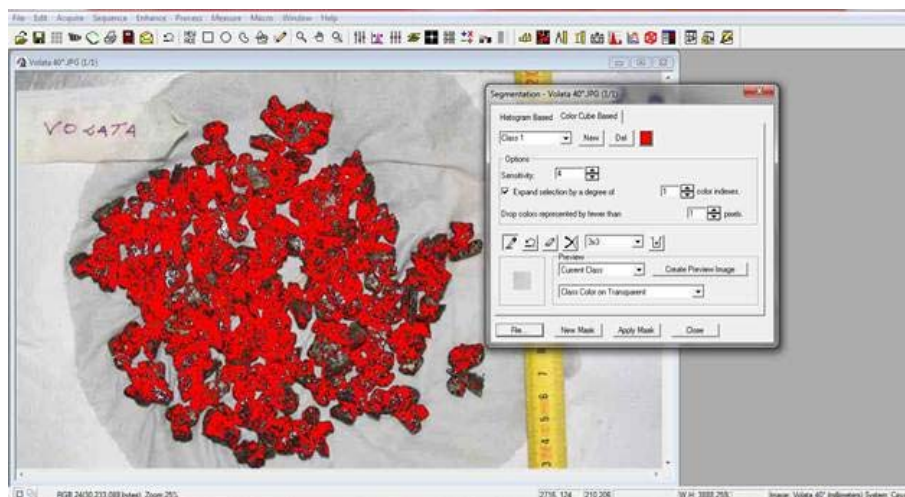
The image analysis technique was used to evaluate stripping test values instead of using visual observation. Analysis was performed on the same samples already subjected to visual observation. As already said when performing stripping tests there must be an evaluation of the percentage of bitumen that has stripped off from the aggregate. In Figure 103 are shown some of the samples on which perform the image analysis technique.



**Figure 103. Before and after performing stripping test**

In order to perform the measurements pictures were taken from different positions of the aggregates and then the analysis was performed. It must be said that this type of image analysis is a 2D static image processes analysis. To perform the analysis on each of the four types of aggregates, first of all the surface area occupied by each of the aggregates shown in each picture was measured. Once this was measured other measurements were performed like measuring the totally black area corresponding to the surface covered by bitumen and also performing measurements on areas representing the uncovered surface by bitumen. Knowing these information's it was possible to calculate the percentage of stripping for each of the aggregate present in a picture. During these measurements it was important not to confuse and measure incorrect areas. For example consider a dark colour mineral of an aggregate as an area covered by bitumen, this was avoided by using filters in order to trace the correct color corresponding to bitumen. In Figure 104 are shown in red the areas of the aggregates covered by bitumen that have been tracked by the software.

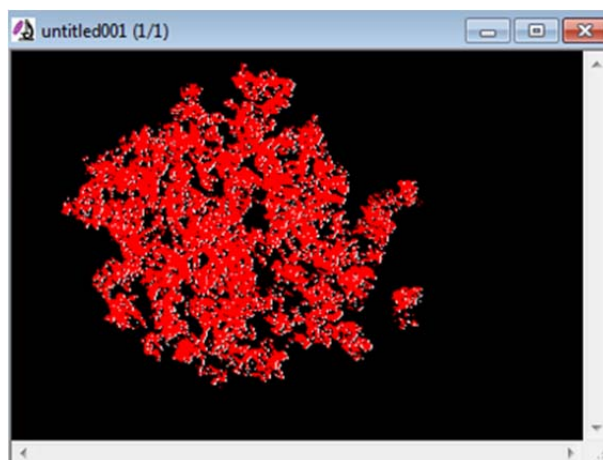




**Figure 104. Image analysis to search black colour**

In

Figure 105 is shown the area covered by bitumen for each aggregate eliminating the rest of the area of the aggregates.



**Figure 105. Area covered by bitumen for each aggregate eliminating the rest**

At this point having calibrated the image in mm we will have the area of stripping as a percentage of the total area occupied by the bitumen on the aggregates. In Table 27 are reported the values obtained by the software for all the four aggregates.

**Table 27. Results from image analysis**

Samples	Test conducted at 25°C Incomplete coating (%)	Tests conducted at 40°C Incomplete coating (%)
Basalt	3	8
Granite from Crusher	5	28
Granite from TBM	46	68
Granite from D&B	12	49

This method once the procedure is established it can be considered simple and fast technique with very little error.

#### **7.4 Possible use of image processing to understand parameters that influence adhesion phenomena**

From the encouraging results obtained using Digital Image Processing (DIP) to evaluate stripping tests values. The study wanted to evaluate the possible application of this method for other purposes. For this reason the possible application of DIP to measure the thickness of the layer of bitumen remained on the aggregate surface after that the stripping tests was conducted was taken into consideration. Also relate this measurement values to parameter of the aggregate like mineralogical composition and geometrical features in order to establish any correlation.

In the present work a procedure is proposed for the determination of such correlation based on performing digital image processing method observing thin sections obtained from samples containing aggregates deriving from stripping tests.

In total n°8 thin sections were prepared divided as follows: two thin sections for each of the four types of aggregates used during the stripping tests that is at 25°C and 40°C. The procedure can be divided in 5 phases: sample preparation, image acquisition, image processing, image measurement and data processing.

#### **7.4.1 Methodology of thin section preparation**

The preparation of thin sections requires experience and ability due to the accuracy that must be reached and the type of machines that have to be used. The thin sections studied in this research were prepared by the technician of the department of DICCAR. Before introducing the procedure of preparation let give a short description of what is a thin section. A thin section is a 30  $\mu\text{m}$  slice of rock attached to a glass slide with epoxy resin. Typical thin section slides are 26\*46 mm, although larger ones can be produced. They are generally covered by another glass slide, a cover slip also attached to the rock with epoxy resin.

This chapter will give an overview of the phases of the preparation. Due to the type of aggregate that we had to deal with that had the surface totally or partially covered by bitumen some arrangements had to be taken to the typical phases of preparation of thin sections.

First of all the bitumen on the surface of the aggregate was sticky and this was a problem when manipulating them. For this reason the aggregate were located in a freezer and left there for 24 h. This made the aggregates easier to handle especially to avoid that the aggregate will stick to the surfaces of the sample holder.

After that the 24 h had passed the aggregates were introduced in silicon sample holders. The samples holders were of a cylindrical shape having the following dimensions 50 mm diameter and 30 mm high Figure 106.



**Figure 106. Silicon sample holders**

Before introducing the aggregates in the sample holders a thin layer of epoxy resin was laid then the aggregates were set in place. The type of resin used was a low-viscosity one and the hardener a long curing time Figure 107. The ratio was two parts of Resin to one part of Hardener by weight.



**Figure 107. Epoxy resin and hardener**

Once the aggregate were introduced a first layer of epoxy resin was slowly pored over the prepared specimen. To make sure that the epoxy resin could penetrate into the voids of the specimens, the sample was embedded under low-vacuum Figure 108.

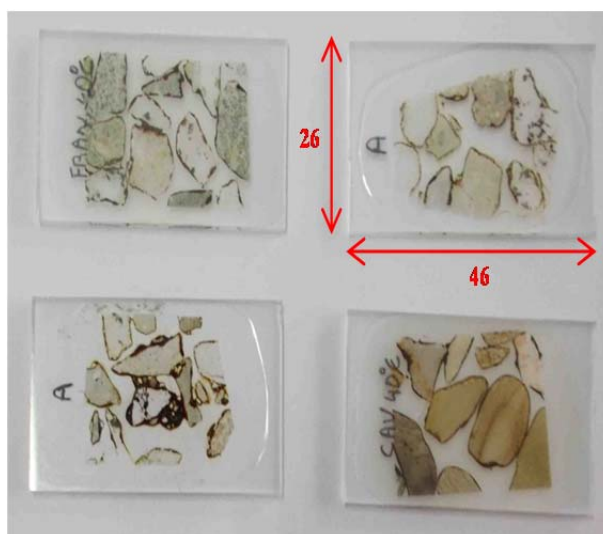


**Figure 108. Specimen under vacuum**

Once the specimen had stabilized and all the voids had been filled a second layer of resin was poured in order to cover totally the aggregates, at this point the sample was once again placed in the low-vacuum. Once the sample had stabilized it was removed from the vacuum and left to harden at a constant temperature of 30°C for 24 h.

After the 24 hours had passed it was possible to remove the specimen from the sample holder. At this point a cylindrical specimen were obtained having the following measures 50 mm diameter and 30 mm high.

Specimens were then reduced in size and shape in order to place them on to a glass slide with the following dimension 26\*46 mm. Cutting was performed using a diamond saw. Once the specimen had been reduced in size and shape, it was glued to the glass slide using epoxy resin and then ground to the correct thickness and polished. This was performed using silicon carbide sandpaper in order to have a smooth and polished surface to capture neat images from microscope analysis. In Figure 109 are shown some of the thin sections prepared to perform digital image analysis.



**Figure 109. Thin sections**

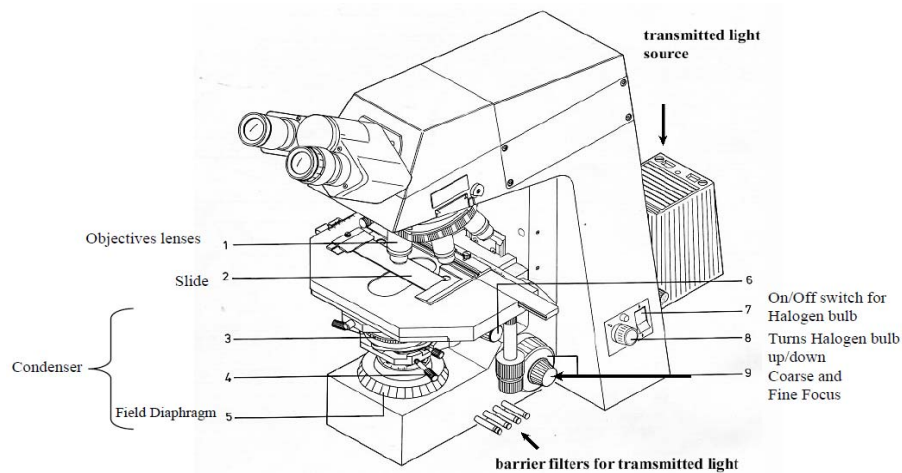
#### 7.4.2 Image acquisition

Once the thin sections were ready, optical light microscopy investigations were carried out with an optical microscope (Axioplan D-7082, Zeiss) in transmission mode with crossed polarizers. A digital camera (PL-A662, Pixelink) connected to a personal computer was used to capture the optical microscopy images see Figure 110.



**Figure 110. Light optical microscope**

In Figure 111 are reported the main features of the Zeiss Axioplan microscope. To perform the investigation the thin section was set in place in the position of the slide (Figure 111, N°2) in order to be crossed by the light. Most important is to adjust the focus to obtain the right focal level. To perform the analysis each thin section was investigated at a magnification of x25, which provided an adequate resolution for the study of the samples.



**Figure 111. Overview of axioplan microscope**

(source Zeiss Axioplan Microscope Instruction Manual)

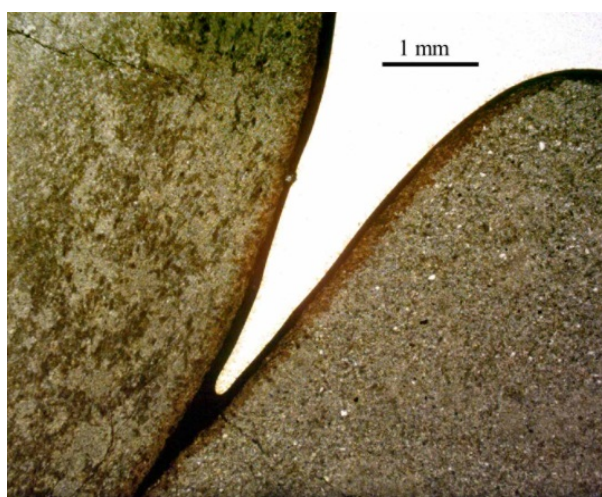


The images were captured using a microscope camera with a resolution of 1280\*1024 pixels Figure 112. The camera was connected to a computer making it possible to view and store images.



**Figure 112. Digital microscope camera**

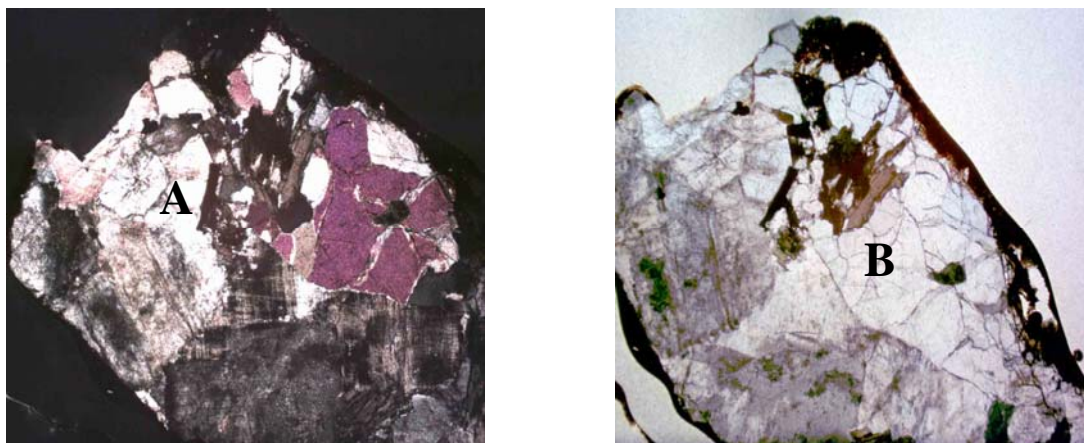
In Figure 113 is shown a portion of a thin section enlarged 25 times with reference line in order to be able to calibrate the image and measure geometrical features when performing image processing. Digital images were recorded with a 1028\*1024 pixels resolution. It is possible to see in Figure 113 the cross section of two aggregates and also the thin layer of bitumen formed on each of the aggregates surface.



**Figure 113. Thin section image enlarged x25**

For each of the thin sections two digital images were obtained using the digital camera connected to the microscope. One image was captured when the thin section was analyzed

in crossed light (Figure 114-A) and the other one with plane-polarized light (Figure 114-B). This procedure was done to distinguish easily the minerals composing the aggregates; for instance, quartz and feldspars are colourless in plane-polarised light, but have distinct interference colours in cross-polarised light.



**Figure 114. Thin section in crossed (A) and plane-polarized light (B)**

The possibility of distinguishing the different types of minerals was important when trying to relate this characteristic to the thickness of the bitumen layer on the surface. For each thin section not all the aggregates present in it were analysed but only those in which both bitumen film layer and mineral composition was well defined. For the correct mineral characterization of the thin sections the supervision of a geologist was needed.

#### **7.4.3 Image processing and measurements**

The imaging procedure that was conducted in order to measure not only the thin layer of bitumen formed on the surface of the aggregates but also relate this value to other characteristics of the aggregates can be summarized by the following steps:

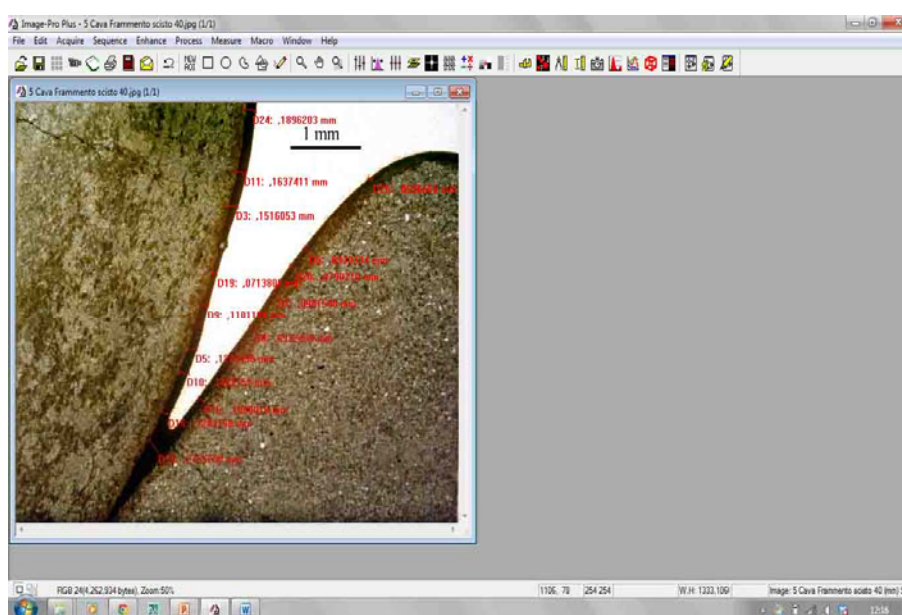
- image calibration was performed in order to fix and measure in mm the parameters of the digital images enlarged by 25 times;
- image thresholding consisting in performing segmentations to extract single objects. Thresholding is an effective tool to separate objects from the background. Many factors such as, ambient illumination, busyness of gray levels within the object and its background, inadequate contrast, and object size not commensurate with the scene, complicate the thresholding operation. In this study difficulties emerged when detecting bitumen layers when in contact with darker minerals. For this reason filters were employed



to increase the difference in contrast between these two objects. Also comparison between the original image in plane-polarized light and the one in crossed polarized light helped to detect the boundaries of the objects;

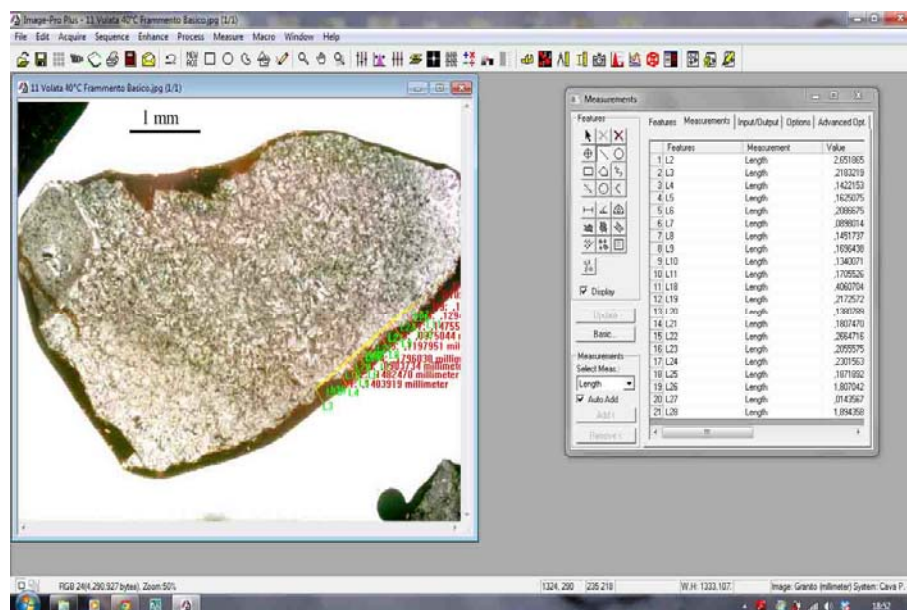
- in some cases automatic count/size process was performed in order to measure the areas of the objects, i.e. aggregate particles, which had high pixel intensity values, against a darker background;

- image measurements were conducted by measuring the film thickness of the bitumen Figure 115, relating it to the surface morphology of the aggregates and also mineral composition.



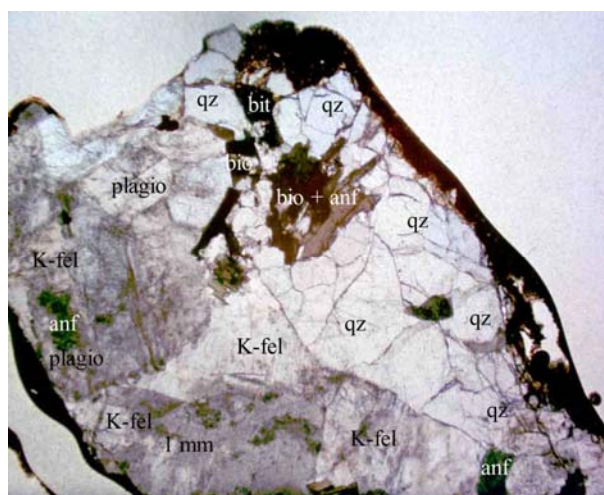
**Figure 115. Bitumen film thickness measurement**

The morphology of the aggregates in this study was intended a maximum roughness depth, measuring peak to valley height Figure 116.



**Figure 116. Progressive measurement of aggregate morphology**

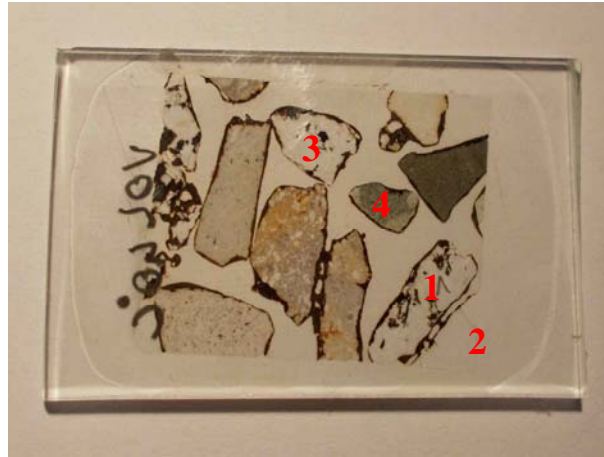
In Figure 117 it is possible to see digital image of the aggregate with the mineral composition.



**Figure 117. Mineral composition of the aggregate**

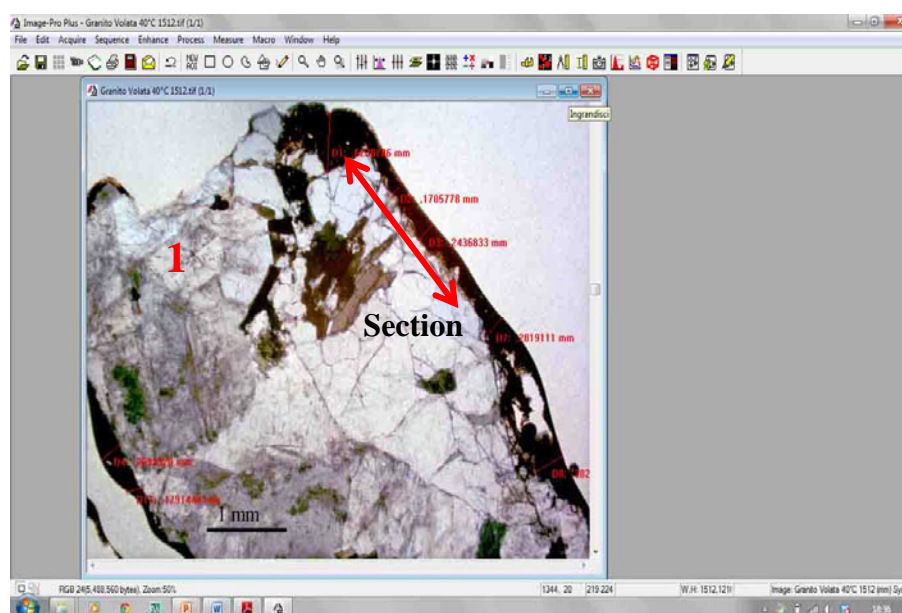
#### **7.4.4 Data processing and results**

Measurements were performed to determine the bitumen film thickness and the roughness of the aggregates in the thin sections. In each thin section aggregates were chosen in order to be analyzed. Figure 118 reports a picture of the thin section in which aggregates deriving from D&B and subjected to stripping tests at 40°C. The aggregates that have been analyzed are marked with red colored numbers (see Figure 118).



**Figure 118. Thin section with aggregates deriving from D&B**

In Figure 119 is shown an enlargement of the aggregate that is present in the thin section in Figure 118 and labeled n°1. Performing microscope analysis and enlarging x25 it possible to see that this aggregate is of a granite type with well definite crystals Figure 117.



**Figure 119. Granite by-product deriving from D&B subjected to stripping test at 40°C**

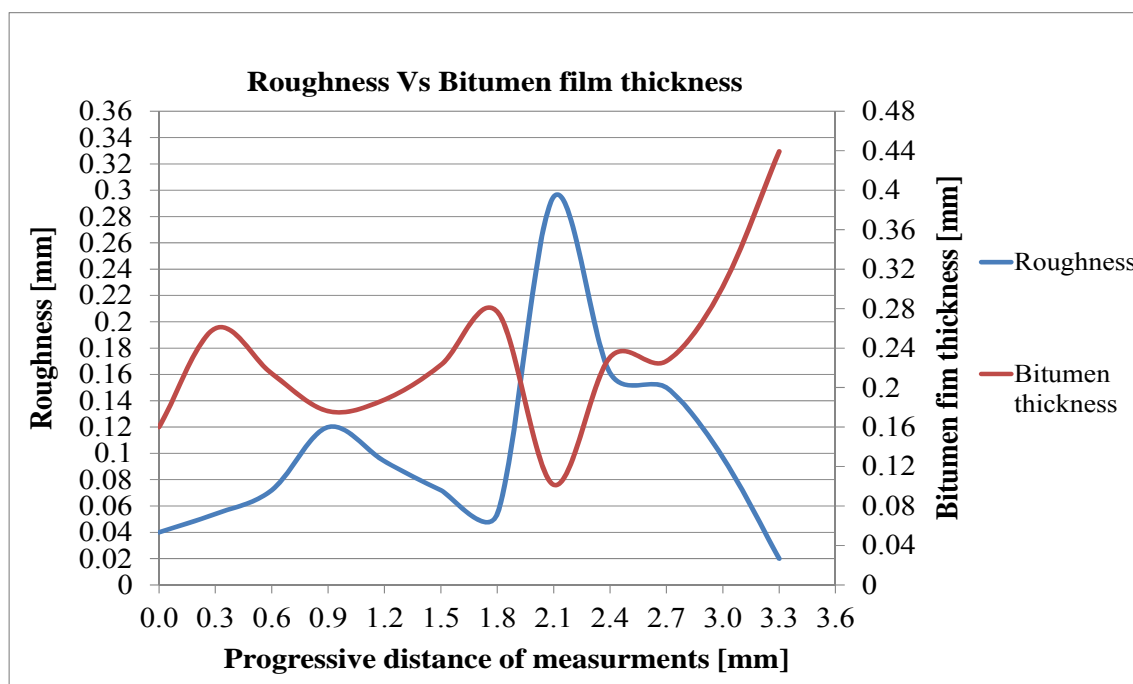
A section was chosen and measurements were performed in order to obtain data on bitumen film thickness and aggregate roughness. Total length of the section was of 3.3 mm with progressive measurements each of 0.30 mm for a total of n°12 points; values are reported in Table 28.

**Table 28. Measurements conducted on the aggregate n°1**

Progressive measurements (mm)	Bitumen film thickness (mm)	Roughness (mm)
0	0.16	0.04
0.3	0.26	0.05
0.6	0.21	0.07
0.9	0.17	0.12
1.2	0.18	0.09
1.5	0.22	0.07
1.8	0.27	0.05
2.1	0.10	0.29
2.4	0.23	0.16
2.7	0.22	0.15
3.0	0.30	0.09
3.3	0.43	0.02

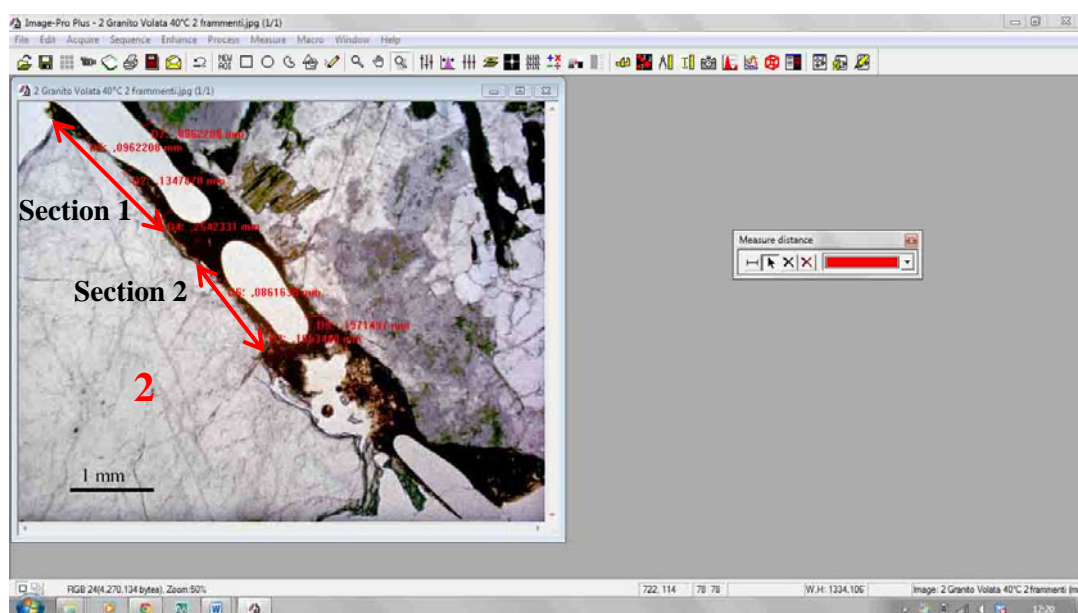
Same procedure was conducted on all the tested aggregates. Always a section was chosen and progressive measurements were performed measuring bitumen film thickness over aggregate surface and aggregate roughness.

In Figure 120 is reported the relation between the roughness of the aggregate and bitumen film thickness. Average roughness of 0.10 mm was measured while film bitumen thickness resulted of 0.23 mm.



**Figure 120. Relation between the aggregate roughness and bitumen film thickness**

Continuing the analyses of the aggregate chosen in the thin section, the aggregate labelled n°2 was analysed. This type of aggregate is also granite with large well defined crystals. In Figure 121 is reported an enlargement of the aggregate. To measure bitumen film thickness and aggregate roughness two sections were chosen; section n°1 length of 1.5 mm and section n°2 with a length of 1.2 mm.



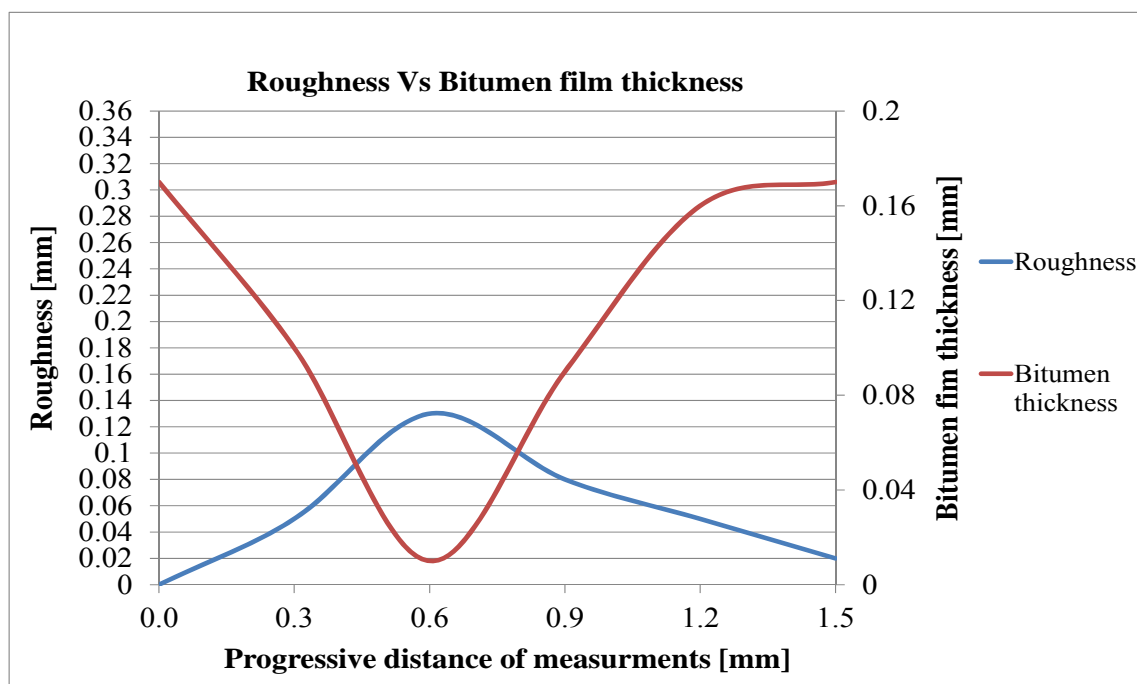
**Figure 121. Granite by-product deriving from D&B subjected to stripping test at 40°C**

In Table 29 are reported the measurements for the section n°1 with progressive measurements of 0.3 mm number of point n°6.

**Table 29. Measurements aggregate n°2 section n°1**

Progressive measurements section n° 1 (mm)	Bitumen film thickness (mm)	Roughness (mm)
0	0.17	0
0.3	0.10	0.05
0.6	0.01	0.13
0.9	0.09	0.08
1.2	0.16	0.05
1.5	0.17	0.02

In Figure 122 is reported the relation between aggregate roughness and bitumen film layer for the values measured in section n°1.



**Figure 122. Relation between the aggregate roughness and bitumen film thickness section n°1**

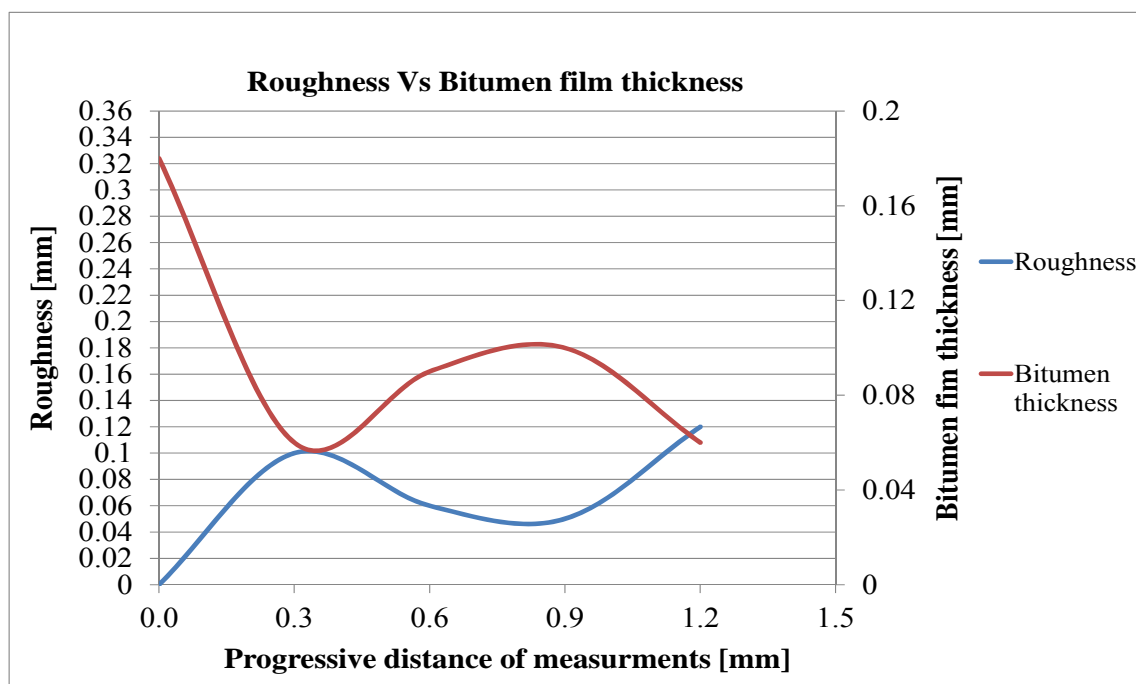
In Table 30 are reported the measurements obtained in section n°2 progressive measurements of 0.30 mm number of point measured in total n°5.

**Table 30. Measurements aggregate n°2 section n°2**

Progressive measurements section n° 2 (mm)	Bitumen film thickness (mm)	Roughness (mm)
0	0.18	0
0.3	0.06	0.10
0.6	0.09	0.06
0.9	0.10	0.05
1.2	0.06	0.12

The chart reported in Figure 123 shows the relation between aggregate roughness and bitumen film layer for section n°2.

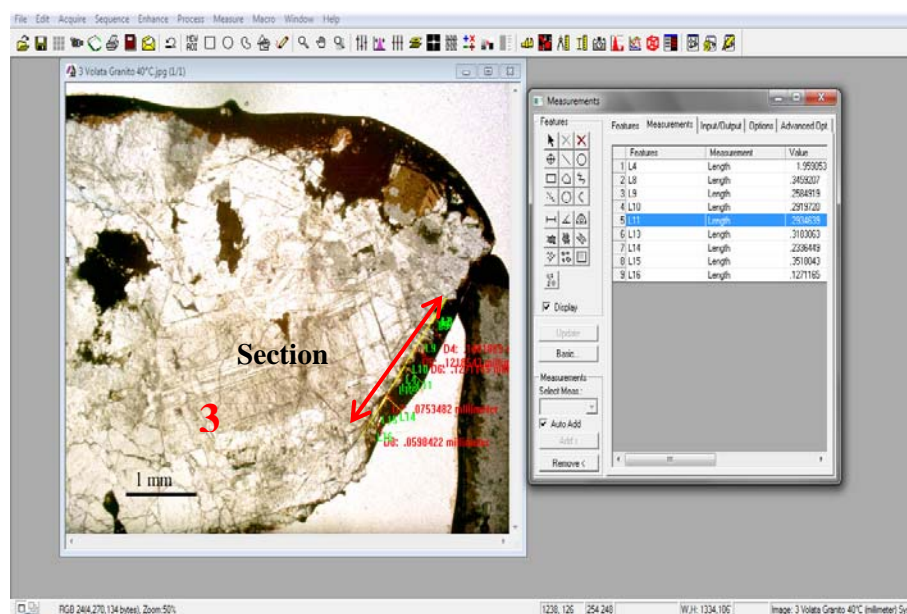




**Figure 123. Relation between the aggregate roughness and bitumen film thickness section n°2**

The average Bitumen film thickness for the two sections was respectively: 0.11 mm for section n°1 and 0.09 for section n°2 while the roughness was respectively 0.05 mm and 0.06 mm. For each of the aggregates shown in the thin sections and subjected to stripping tests, measurements were performed in order to obtain aggregate roughness and bitumen film thickness. In Figure 124 aggregate labeled n°3 was analyzed, also this type of aggregate was granite, the section chosen to be analyzed showed higher values of roughness.





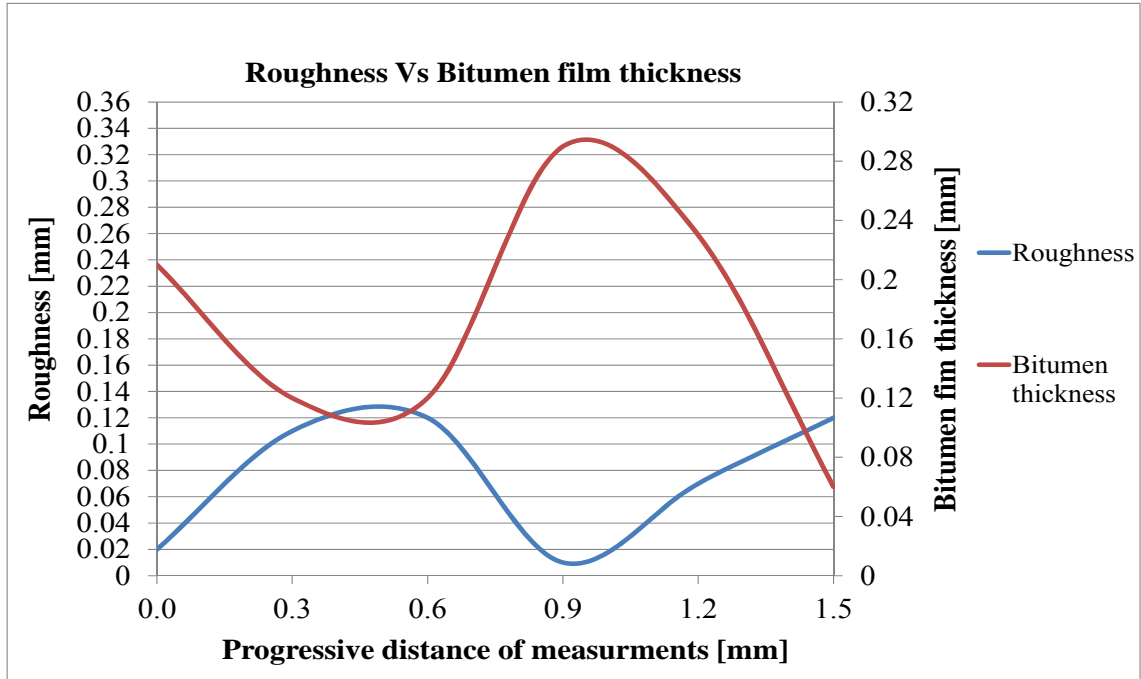
**Figure 124. Granite by-product deriving from D&B subjected to stripping test at 40°C**

Results obtained during the measurements are reported in Table 31 total length of the measured section was of 1.5 mm total measured points n°6.

**Table 31. Measurements of aggregate labelled n°3**

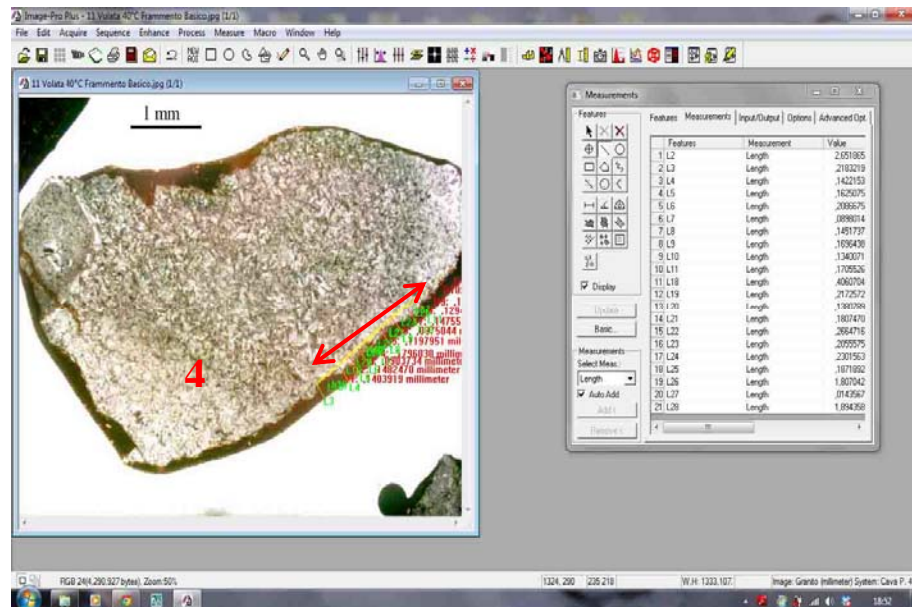
Progressive measurements section n° 2 (mm)	Bitumen film thickness (mm)	Roughness (mm)
0	0.21	0.02
0.3	0.12	0.11
0.6	0.12	0.12
0.9	0.29	0.02
1.2	0.23	0.07
1.5	0.06	0.12

The values of film thickness and aggregate roughness are reported in the chart shown in Figure 125.



**Figure 125. Relation between the aggregate roughness and bitumen film thickness aggregate labelled n°3**

The last aggregate that was analysed in the thin section was the aggregate labelled n°4. This type of aggregate resulted of a fine grained deriving from a basic rock Figure 126.



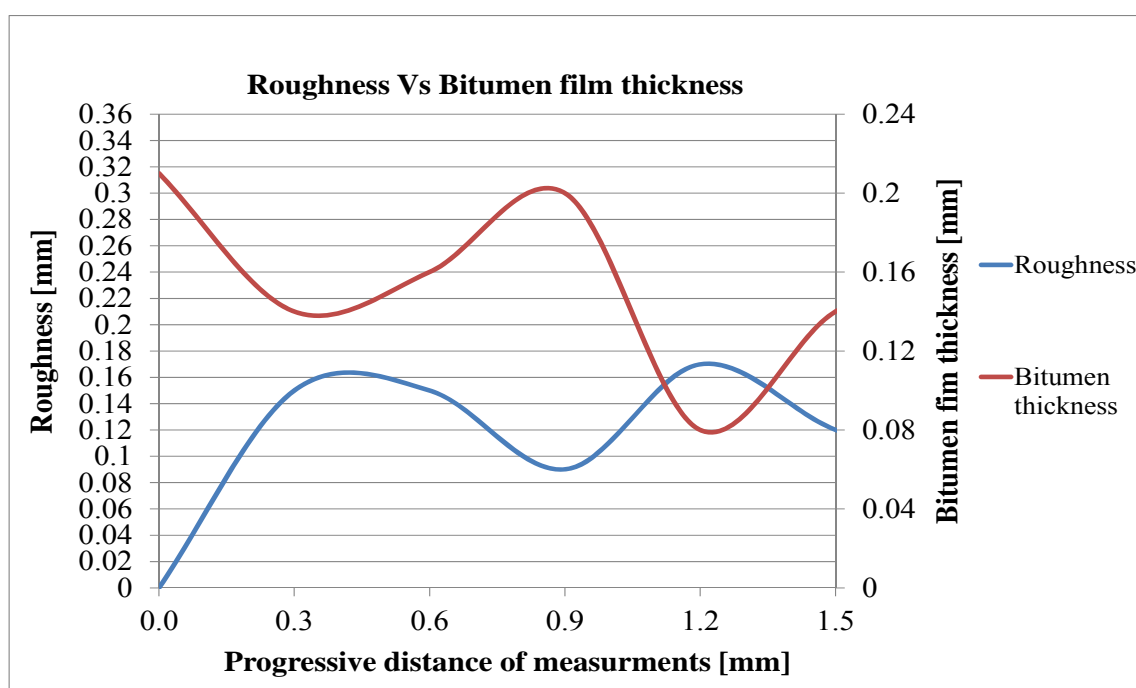
**Figure 126. Basic rock deriving from D&B subjected to stripping test at 40°C**

The measurements performed on the aggregate shown in Figure 126 are reported in Table 32. Progressive measurements were performed every 0.30 mm for a total of n°8 measuring points.

**Table 32. Measurements of aggregate labelled n°4**

Progressive measurements [mm]	Film Thickness [mm]	Roughness [mm]
0	0.21	0
0.3	0.14	0.15
0.6	0.16	0.15
0.9	0.20	0.09
1.2	0.08	0.17
1.5	0.14	0.12
1.8	0.16	0.09
2.1	0.17	0.12

The values reported in Table 32 were used to obtain the chart reported in Figure 127.



**Figure 127. Relation between the aggregate roughness and bitumen film thickness aggregate labelled n°4**

## **8. CRITICAL DISCUSSION AND CONCLUSIONS**

The following chapter will focus the attention first of all on evaluating if the aims proposed by the research have been achieved. Considering the potential applications of granite by-products to be used in the road industry, it is possible to state that the test conducted on Sardinian granite by-products, deriving from the Sarrabus-Gerrei region, have shown that they can be considered a valid alternative to the aggregates that are currently used in Sardinia. Focusing the attention of their use in all the pavement layers it must be said that there are some critical issues.

Considering the evaluation of verifying if different processes to which granite is subjected can influence the physical and mechanical behavior for its use in road pavement layers, tests have shown that granite by-products in some cases behave differently considering the nature of the process. Of the three granite by-products tested each of them needs further processes. Crushing and screening must be performed but also washing each of the obtained size fractions must be performed. In the following subchapters specific objectives proposed will be discussed.

### **8.1 Environmental compatibility**

The tests conducted on the three types of granite by-products in order to establish the environmental compatibility have shown that in some cases the processes to which these materials are subjected can contaminate them. The results obtained from the leaching tests have proven that aggregates AG-2 and AG-3 have a concentration of Fluoride (F) over the concentration limit (1.5 mg/l). The aggregates AG-2 were well over the concentration limit while aggregates AG-3 just slightly over. This can be attributed to the type of explosive used during the excavation phase. In fact the type and amount of explosive used was able in some cases to contaminate the aggregates during the explosion. For this reason the washing of the aggregates could be considered in order to reduce such contamination inside the imposed concentration limit. Also it has been possible to establish that the pH values that were measured for AG-2 samples showed that they were always in the range of pH 12, while AG-1 was in the range of pH 10 and AG-3 pH 9.6.

Values of pH like the one obtained for AG-2 aggregate have been measured when performing leaching tests on granite fines deriving from gang saw (Delgado et al., 2005).

## **8.2 Unbound layers**

Test conducted on the three types of granite by-products to evaluate the possible application for unbound layers have shown that first of all the process to which they are subjected can influence the grading but also the shape. Following the results from the particle size distribution it is possible to establish that aggregates deriving from TBM have a content in fines that is in the range of 10% more in respect to the are two types of aggregates deriving that is: D&B and from mobile crusher. Also the values of standard deviation for the particle size distribution show that AG-1 is the aggregate that has a variation that is always in the range of 2%, while aggregates AG-2 is the aggregate in which there is wide range of variation for each of the size fractions, this is due to the method of excavation that produces a more ether-genus material with variable size fractions. Analyzing the values obtained from the modified Proctor test it is possible to see that these values can be related to the particle size distribution of the three aggregates. AG-1 in which optimum water content values has an average value of 6.5% while dry density is  $21.55 \text{ kN/m}^3$ , this can be related to the presence of more fines, that not only absorbing more water they tend to fill the voids in the coarse aggregate acting as workability agents improving dry density values. Also the higher values obtained performing the Los Angeles abrasion tests on AG-1 aggregates can be explained by the shape; flat and elongated that tends to disaggregate more easily.

Following the results from the Atteberg limit test the three types of aggregates resulted non plastic. The sand equivalent value for AG-2 and AG-3 didn't achieve the minimum value of 40% for foundation layers. Considering the test conducted to determine Californian bearing ratio the values obtained are in the range of 150% and 180% these are good values. Following the results from the tests conducted it is possible to establish that first of all the three granite by-products if they are to be used in foundation and base layers they need to be crushed and screened in order to have the correct fractions to achieve the specified blend. Also the crushing will be useful especially for aggregate AG-1 in order to modify the shape of the aggregates to achieve better values when performing the Los Angeles abrasion test. Overall more specified tests are needed in order to be able to better

understand the possible applications of this type of material in unbound granular pavement layers.

### **8.3 Hydraulic bound layers**

The test conducted on the cement bound granular specimen have shown according to the C.N.R. 29/72 specifications that of the two types of granite by-products tested (AG-1 and AG-2) showed excellent values of compressive strength with low content of cement. The value imposed by the specifications is between 2.45 MPa and 4.41 MPa. The minimum compressive strength value inside the specified range, for aggregates AG-1, has been of 3.28 MPa with cement content of 2% this is a good achievement knowing that the percentage of cement permitted is up to 5%. Results of AG-2 aggregate, showed that the minimum strength value inside the specified range was of 2.46 MPa with cement content of 2%. The results obtained showed how this type of aggregate can achieve good values of compressive strength with low percentages of Portland cement. In order to use these types of materials the aggregates must be crushed to obtain the correct size fractions.

### **8.4 Affinity between bitumen and granite by-products**

Evaluating the specimens obtained by mixing AG-2 aggregate with bitumen and filler and then compacting using a gyratory compactor, it has been possible to establish that there are cohesion problems between granite and bitumen. The first set of tests a grading for HMA base layers was prepared using as a filler granite fines. In order to verify if the cohesion problem was due to the filler used, a second set of tests was performed this time using an open-graded HMA layer. Also in this case cohesion problems appeared. For this reason stripping tests were performed to better evaluate the nature of the problem. The results obtained showed how granite aggregates don't have any affinity with bitumen, this is widely stated and depends on the nature of bitumen and granite aggregates. For this reason in order to achieve good values of cohesion, adhesion promoters were used when preparing the mix. The results obtained showed how with a very small percentage of adhesion promoters the stripping was reduced by 75%.

From these results it was possible to establish that the use of adhesion promoters can reduce the stripping value in the range of the imposed limit. The use of this type of additives although they are very effective in reducing the de-bonding they do influence the cost of the infrastructure.

## **8.5 The use of digital image analysis**

The introduction of digital image analysis to better evaluate the results of the stripping tests can be considered a methodology that can improve the reliability of the results being more objective and rapid. For this reason the digital image processing method used to perform the analysis had to be calibrated in order to evaluate the type of error that was being obtained during the measurements. From the results obtained it can be said that image analysis can be a valid support to perform such measurements. Using this type of technique can improve the understating of parameters that can influence cohesion but also be able to measure them in a fast and simple way. The results obtained by analyzing the images of the thin sections have shown that the roughness of the surface of the aggregates does influence the thickness of the bitumen layer. Also from the tests conducted while granite aggregates show a more wide range of bitumen film thickness, in basic rocks like basalt the bitumen film thickness is more uniform with average values lower than of those achieved on granite. This can be explained by the roughness of the aggregate but also of the nature of the aggregates if considering the grained composition. Granite has a coarse grained composition while basalt has a more fined one. The possibility of establishing if there is a relation between different mineral compositions that form the aggregate surface and the bitumen film thickness at this stage can't be stated due to the number of tests performed. What can be said is that the possibility of analyzing thin section in which aggregates are covered by bitumen, using image analysis can be a valid instrument to conduct such measurements.

## **9. RECOMMENDATIONS FOR FURTHER RESEARCH**

The results from this work cannot be used directly as guidelines to use Sardinian granite by-products in road pavement layers. However, some of the results may be useful for further studies to validate rules and quality control methods to establish critical factors.

It is recommended that further case studies should be performed in order to have an overall picture, knowing that in Sardinia there are several types of granite. For this reason more material should be tested deriving from different granite quarries. In the specific while a major part of the granite by-products lie unbounded in stockpiles and hasn't been yet subjected to major climate factors, there is also another type of granite named decomposed granite that has weathered to the point that it fractures in smaller pieces, also this type of material should be tested.

- More tests should be performed in order to evaluate the behaviour of such materials in unbound road pavement layers. Following the results obtained from the tests and verifying that in some of them the results obtained didn't reach the values imposed by the specifications, it would be interesting to evaluate how these aggregate behave in real working conditions performing field testing on a trial road section.
- More tests are needed to study the behaviour of cement bound granular layers. In the specific perform tests in order to understand if the good compressive strength values obtained with low percentage of cement can be related to a cementing chemical behavior performed by the granite fines.
- Considering the cohesion problem between granite and bitumen, it would be interesting to evaluate the possibility of achieving good values of stripping replacing the adhesion promoter used in this research and replacing it with hydrated lime. Tests will have to focus the attention on evaluating if hydrated lime can act as a potential adhesion promoter when used with granite aggregates but also evaluate if it can be used as a filler. With the final achievement of using one product for two scopes.



## REFERENCES

- ACI Committee. (1997). State of the Art Report on Soil Cement. *American Concrete Institute*.
- Akbulut and Gurer. (2007). Use of aggregates produced from marble quarry waste in asphalt pavements. *Building and Environment*.
- Barksdale. (1972). Laboratory Evaluation of Rutting in Base Course Materials. *3rd International Conference on the Structural Design of Asphalt Pavements*. London.
- Barksdale and Itani. (1989). Influence of aggregate shape on base behavior. *Transportation Research Record*.
- Barksdale et al. (1991). Measurement of aggregate shape, surface area, and roughness. *Transportation Research Record*, pag.107-116.
- Berthelot et al. (2010). Mechanistic characterization of cement stabilized marginal granular base materials for road construction.
- Bikerman. (1961). The Science of Adhesive Joints. *Academic Press*.
- Binici et al. (2008). Durability of concrete made with granite and marble as recycle aggregates. *journal of materials processing technology*.
- Bons and Jessel. (1996). Image analysis of microstructures in natural and experimental samples. *Pergamon*.
- Bralia et al. (1982). Aspetti genetici del batolite ercinico Sardo. *Rend . Soc. Miner. Petr.*
- Brotzu et al. (1993). The search for the parental magma of the high-K calcalkaline rock-serie in the southrenmost Sardinia Batholith, pag.253-280.
- Bruno et al. (2011). Image analysis for detecting aggregate gradation in asphalt mixture. *Construction and Building Materials*.
- Carosi et al. (2006). The variscan basement of northern sardinia (Italy): Field guide to the excursion in the baronie region., Pisa, pag.13-29.
- Chandra and Choudhary. (2012). Granite and marble dusts as filler in asphalt concrete.
- CNR-UNI 10014. (1969). Determination of the Consistency (Atterberg) of a Soil. *Italian National Research Council (CNR) and Italian Organization for Standardization (UNI)*.

- Daučík et al. (2011). Methods for determination of affinity between aggregate and bitumen.
- Dawson. (1994). Prediction of in-situ mechanical performance of unbound aggregate, is it possible. *Annual Symposium, College Station, Texas*.
- De Leonardis. (2011). The legal provisions governing waste in the. *Copyleft – Ius Publicum*.
- Delgado. (2005). Geochemical assessment of the contaminant potential of granite fines produced during sawing and related processes associated to the dimension stone industry. *Journal of Geochemical Exploration, Elsevier, pag.25-27*.
- Directive 2008/98/ Ec of the european parliament and of the council. *Official Journal of the European Union*.
- Deryaguin and Smilga. (1969). Adhesion: Fundamentals and Practice. *London: Ministry of Technology*.
- Dhanapandian and Gnanavel. (2010). Using Granite and Marble Sawing Power Wastes in the Production of Bricks: Spectroscopic and Mechanical Analysis. *Research Journal of Applied Sciences, Engineering and Technology*.
- European Comission. (2012). Green Public Procurement a collection of good practices. *Publications Office of the European Union*.
- Founti et al. (2010). Environmental management aspects for energy saving in natural stone quarries.
- Fuente. (1997). A computer-controlled rotating polarizer stage for the petrographic microscope, pag.203-208.
- Gattacceca et al. (2007). Miocene rotation of Sardinia: New paleomagnetic and geochronological constraints and geodynamic implications. *Earth Planet. Sci. Lett.*, pag.359-377.
- Gazi et al. (2012). Energy efficiency and environmental assessment of a typical marble quarry and processing plant.
- Ghezzi et al. (1972). Lineamenti geopetrologici del cristallino sardo. Le plutoniti e la loro successione negli eventi intrusivi., pag. 205-234.
- Hall. (1998). Testing protocols to evaluate performance potential of unbound granular pavement layers.

- Hecht. (2000, 2004). Apollonian packing and fractal shape of grains improving geomechanical properties in engineering geology. *In Pure and Applied Geophysics*, pag.157.
- Hicks and Monismith. (1971). *Highway Research Record*, pag.15-31.
- Hicks and Monismith. (1971). Factors Influencing the Resilient Response of Granular Materials. *Highway Research Record*, pag.15-31.
- Hunter et al. (2004). Effect of asphalt mixture compaction on aggregate orientation and mechanical performance. *8th Conference on asphalt pavements for Southern Africa*.
- Jagan et al. (2006). AIMS: The Future in Rapid, Automated Aggregate Shape and Texture Measurement.
- Kezdi. (1979). Stabilized earth roads, developments in geotechnical engineering. *New York: Elsevier Scientific Publishing Company*.
- Li. (2005). Application of acidic aggregates in asphalt concrete pavement. 138-139.
- Li et al. (1993). Quantitative analysis of aggregate shape based on fractals. *ACI Mater*, pag.357-365.
- Mac Bain and Hopkins. (1925). On adhesives and adhesive action. *Journal of Physical Chemistry*, pag.188-204.
- Marinoni et al. (2004). Characterisation of mortar morphology in thin sections by digital image processing. *Cement and Concrete Research*.
- Masad. (2004). Aggregate Imaging System (AIMS) Basics and Applications. *Texas Department of Transportation and Federal Highway Administration*.
- Myers. (1991). Surfaces, interfaces, and colloids: Principles and applications. *VCH Publishers*.
- Natural stones from Sardinia, *Book*. (2002).
- Official Journal of the European Union. (2008). Directive 2008/98/ec of the European parliament and of the council. European Union.
- Pepino. (2009). Pepino, M. (2009). Scavo meccanizzato di gallerie: previsione della qualità del marino nell'ottica del suo riutilizzo.
- Perez et al. (2013). Dredged sediments management in Italy: legal aspects. *International Association of Dredging Companies*.

- Petersen et al. (1982). Chemistry of asphalt-aggregate interaction: Relationship with moisture damage prediction test. *Transportation Research Record*.
- Pocius. (1997). Adhesion and adhesives technology. *Gardner Publications, Inc.*
- Quarry office. (2007). *Sardinian regional government*.
- Raad et al. (1992). Characterization of saturated granular bases under repeated loads. *Transportation Research Record*.
- Rada and Witczak. (1981). Comprehensive evaluation of laboratory resilient moduli results for granular material. *Transportation Research Record*, pag.23-33.
- Regione Autonoma della Sardegna. (2012). Linee guida per l'applicazione degli acquisti pubblici ecologici negli appalti di lavori: utilizzo degli sfridi delle cave di marmo e granito nel settore delle costruzioni stradali.
- Ribeiro et al. (2008). Uses of aggregates produced from marble and granite quarry waste in asphalt pavements: a form of clean technology.
- Schultz and Nardin. (2003). Theories and Mechanisms of Adhesion. France: *Centre de Recherches sur la Physico-Chimie des Surfaces Solides*.
- Secchi and Lorrai. (2001). Some geological and environmental aspects of the Sàrrabus-Gerrei Region (SE Sardinia, Italy). *Rendiconti Seminario Facoltà Scienze Università Cagliari Supplemento*.
- Secchi and Lorrai. (2001). Some geological and environmental aspects of the Sàrrabus-Gerrei Region (SE Sardinia, Italy). *Rendiconti Seminario Facoltà Scienze Università Cagliari Supplemento* Vol. 71 Fasc. 2.
- Semmelink. (1991). The effect of material properties on the compactability of some untreated roadbuilding materials. *Department of Civil Engineering University of Pretoria*.
- Sharp. (1990). The Penguin dictionary of chemistry. *Penguin Books Ltd*.
- Sharpe and Schonhorn. (1963). Theory given direction to adhesion work: new theory is based on surface energetics, helps predict what constitutes a good adhesive. *Chem. Eng. News*, pag.67-68.
- Simplicio et al. (1974). Il metamorfismo e il magmatismo paleozoico della Sardegna., pag. 979-1068.
- Tarrar and Wagh. (1992). The effect of the physical and chemical characteristics of the aggregate on bonding. Washington, D.C.: *Strategic Highway Research Program*.

- Thom and Brown. (1988). The effect of grading and density on the mechanical properties of a crushed dolomitic limestone. *Proceedings 14th ARRB Conference*.
- Thompson and Robnett. (1979). Resilient properties of subgrade soils. *Transportation Engineering Journal*, pag.71-89.
- UNI-EN 1097-2. (2010). Tests for mechanical and physical properties of aggregates. *Italian Organization for Standardization (UNI)*.
- UNI-EN 12697-11. (2012). Determination of the affinity between aggregate and bitumen. *Italian Organization for Standardization (UNI)*.
- UNI-EN 12697-31. (2007). Specimen preparation by gyratory compactor. *Italian Organization for Standardization (UNI)*.
- UNI-EN 13286-2. (2010). Test method for laboratory dry density and water content. *Italian Organization for Standardization (UNI)*.
- UNI-EN 13286-47. (2006). Test method for the determination of Californian bearing ratio (CBR), immediate bearing index and linear swelling. *Italian Organization for Standardization (UNI)*.
- UNI-EN 933-1. (2009). *UNI EN 933-1*, Tests for geometrical properties of aggregates. *Italian Organization for Standardization (UNI)*.
- UNI-EN 933-8. (2000). Tests for geometrical properties of aggregates Assessment of fines Sand equivalent test. *Italian Organization for Standardization (UNI)*.
- Uthus. (2007). Deformation Properties of Unbound Granular Aggregates. *Department of Civil and Transport Engineering Norwegian University*.
- Van Niekerk. (2002). Mechanical Behavior and Performance of Granular Bases and Subbases in Pavements. *Road and Railway Engineering*.
- Voyutskii. (1963). Autohesion and Adhesion of High Polymers. *Wiley-Interscience*.
- Yue and Morin. (1996). Digital image processing for aggregate orientation in asphalt concrete mixtures. *Can J Civil Eng*, pag.480-489.
- Zhou. (2003). Analysis of aggregate properties which related to adhesion. pag.19-24.